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CONTENTS

(Cu_{0.4}Al_{0.3})TaSe₂: PREPARATION AND CRYSTAL STRUCTURE ANALYSIS FROM X-RAY POWDER DIFFRACTION

GRIMA-GALLARDO, Pedro^{1,2,3}; DURÁN, Sonia⁴; MUÑOZ, Marcos⁴; RAI, Dibya P.⁵; DELGADO, Gerzon E.⁶

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ABSTRACT

A new phase of the (CuAlSe₂)_{1-x}(TaSe)_x alloy system was synthesized by the melt and annealing technique and studied by SEM, DTA, and XRPD techniques. Its structure has been refined by the Rietveld method using X-ray powder diffraction data. The new alloy corresponds with the stoichiometry Cu_{0.4}Al_{0.3}TaSe₂. This compound crystallizes in the hexagonal space group $P\bar{6}m2$ (Nº 187) with a MoS₂-type structure, and unit cell parameters $a = 3.455(2)$ Å, $c = 13.423(4)$ Å, $V = 138.7(1)$ Å³, $Z = 2$. The crystal structure is based on the MoS₂-type of stacking of TaSe₂ layers with a partial ordering of Cu and Al cations over the tetrahedral sites. The powder pattern was composed of 63.1% of the principal phase Cu_{0.4}Al_{0.3}TaSe₂ and 29.9% of CuAlSe₂, 7.0% of TaSe₃, as the secondary phases

Page - 1

REVIEW ABOUT DIABETES MELLITUS AND URINARY TRACT INFECTIONS

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ABSTRACT

Diabetes mellitus (DM) is a clinical disease correlated with a deficiency of insulin secretion or action. It is one of the leading causes of morbidity and mortality worldwide. The global burden of diabetes is rising due to increasing obesity and population aging. Urinary tract infections (UTI) are common microbial infections known to affect the different parts of the urinary tract accounting for major antibacterial drug consumption. About 150 million UTI cases were diagnosed every year. Urinary tract infections are the most important and most common site of infections in a diabetic patient. Diabetic patients have been found to have a 5-fold frequency of acute pyelonephritis at autopsy than non-diabetics. Most of the urinary tract infections in patients with diabetes are relatively asymptomatic. The presence of this syndrome predisposes to much more severe infections, particularly in patients with acute ketoacidosis, poor diabetic control, diabetic complications such as neuropathy, vasculopathy, and nephropathy. The Gram-negative aerobic bacilli are the large group of bacterial pathogens that cause UTI with few species of Gram-positive bacteria. However, some fungi, parasites, and viruses have also been reported to invade the urinary tract. Urinary tract infection affects women more than men due to several factors such as proximity of the genital tract to the urethra, anatomy of the female urethra, sexual activity, menopause, and pregnancy. Other possible risk factors of UTI include allergy, obesity, diabetes, past history of UTI, contraceptive use, catheter use, and family history.

Page – 07

INVESTIGATING THE EFFECT OF COLD TEMPERATURE STRESS ON UNOPENED MALE CATKINS AND INOCULATED FEMALE FLOWERS OF IRANIAN NATIVE HAZELNUT CULTIVARS

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ABSTRACT

In many low-temperature areas, the environmental factor is an important limiting factor for the production and distribution of horticultural plants. This study aimed to investigate the cold tolerance of the male catkins and inoculated female flowers to screen the popular native hazelnut cultivars in Qazvin under low-temperature stress. A completely randomized factorial block design with three replications was used in this experiment with eight cultivars (Nakhnroud, Khandan, Mish-Pestan, South of Qarabagh, Asl-e-Qarabagh, Rasmi, and Gerdashkevar). After removing each of the treated samples at the end of the experiment, the samples were examined morphologically (appearance) and compared with the control. The changes were recorded as qualitative traits. To understand the influence of cold stress on reproductive organs, hydrogen peroxide and proline were measured. The results showed the onset of freezing in unopened male catkins at -7 and -9 °C and in inoculated female flowers at -3 °C. Damage to unopened male catkins' tissue occurred at -11 °C and in female flowers at -5 °C. The highest value observed among cultivars in the case for proline content of male catkins was in Mish-Pestan and Khandan cultivars with 0.816 and 0.660 $\mu\text{mol/g FW}$, respectively. In inoculated female flowers, Mish-Pestan and Tabestaneh cultivars with 0.185 and 0.168 $\mu\text{mol/g FW}$, respectively, showed the highest statistically significant increase in proline content. Interestingly, the cultivars with the highest proline content in male catkins indicated the most increase in H₂O₂; Mish-Pestan and Khnadan with 0.569 and 0.541 ug/g FW , respectively. Asl-e-Qarabagh was observed to have the least H₂O₂ content (0.042 ug/g FW) among cultivars. Again, in inoculated female flowers, those with the highest concentration of proline (Mish-Pestan and Tabestaneh) were found to have the highest H₂O₂ content (0.335 and 0.331 ug/g FW , respectively).

Page - 17

CALCULATION FOR REDEMPTION OF COMPACT TESTING BY THE PROCTOR METHOD THROUGH NEWTON'S GRAVITATIONAL POTENTIAL ENERGY

FIDELIZ, Fábio Vinícius^{1*}; SILVA, Thiago Augusto Andrade da¹; FERREIRA, Mariana Babilone de Souza¹

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ABSTRACT

Throughout this article, a study on the characteristics of the compaction test by the Proctor Method, regulated by ABNT NBR-7182, which is used to verify the degree of soil compaction, will be approached in order to broaden the discussion and raise points that demonstrate the urgent need to make it more accurate, efficient and safe. Through qualitative and quantitative research carried out by the authors of this article, it sought to collect data through a questionnaire for professionals in the field of geotechnics in the "Quadrilátero Ferrífero" region in Minas Gerais. In addition to other relevant data for the topic, it was raised that of the 22 professionals from the participating region, 72.7% of the total belief that the manual compaction test can be manipulated by an operator during the test execution, failing to generate results reliable, thus showing the importance of the proposed theme. In this way, we initially sought to correlate the Compaction Energy formula idealized by Ralph Proctor with Isaac Newton's Gravitational Potential Energy formula and, through it, present the resizing, which may enable the construction of manual, semi-automatic human propulsion machines (not or making the automated ones that depend on electricity available to the market. In conclusion, from the mathematical calculations, it was possible to evidence the use of Newton's Gravitational Potential Energy to construct new equipment to carry out this test.

Page - 28

EXTRACTION AND CHARACTERIZATION OF CURCUMIN FROM TURMERIC RHIZOMES GROWN IN MÉRIDA, VENEZUELA

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ABSTRACT

The extraction of naturally occurring compounds is one of the fastest-growing industries because of its benefits against its synthetic analogs. Environmental protection must require the use of natural products instead of chemicals to minimize pollution. Thus, this investigation studies the use of some natural product, as curcumin, as naturally occurring acid-base indicators. Curcumin can be used as acid-base indicators since it was found that it possesses pH-dependent solubility. Curcumin, the major active component of turmeric, *Curcuma longa* (Zingiberaceae), is used as a spice in curry and as a coloring agent in yellow mustards, cosmetics, pharmaceuticals, and hair dyes. In this research, the main compound colored rhizome of turmeric (*Curcuma longa*) cultivated in Mérida, Venezuela, is extracted: Curcumin (C₂₁H₂₀O₆) (1E,6E)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione, in a yield of 3.42% after 8 hours of extraction using soxhlet extractor system with organic solvents (hexane and ethanol). The thin-layer chromatography and column performed separation and purification using a mobile phase, a mixture of chloroform-hexane 3:2. The dye was characterized by spectroscopic analysis of visible ultraviolet (UV-Vis) and infrared (IR), in addition to his studio in steering sensitivity as an acid-base indicator. This dye is useful as an acid-base indicator in strong acid-strong base volumes and did not require large amounts of it as it has high sensitivity. The results indicate that curcumin as an acid-base indicator allows the development of new standards in different chemistry fields that require this type of analysis.

Page – 37

THERMAL AND ECONOMIC ANALYSIS OF LIME PRODUCTION

DA ROSA, FLÁVIA SAPPER¹; LHAMBY, ANDRESSA¹; GUILLET, VINICYUS MOURÃO MONTEIRO¹; KLUNK, MARCOS ANTÔNIO²; CAETANO, NATTAN ROBERTO³

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ABSTRACT

The ore beneficiation process uses little technology, but mining companies have high economic performance. As an energy source, firewood plays a fundamental role due to its simple storage, low cost, great availability of forests, and lack of processing. Thus, the present work aims to analyze the consumption of firewood as fuel and possible improvements in the process for the production of lime in terms of harnessing raw materials and costs. Calcium oxide is obtained from the thermal decomposition of calcium and magnesium carbonates obtained from dolomitic deposits of limestone (CaCO₃ : CaMgCO₃). After CaO extraction, it is subjected to a calcination process, removing carbon dioxide (CO₂) in ovens that work at temperatures between 900 and 1200 °C. The source of energy applied to the calcination furnaces in the analyzed area is wood. The wood has a calorific value between 2,250 and 2,700 Kcal/Kg, but the moisture content responsible for the thermal variation must be considered. The firewood burning process was carried out in a ravine type oven where the temperature at which operators are exposed to heat was evaluated. The results indicated that the cooking time dropped by 20% as the amount of wood is fed into the oven. This increase represents a significant gain in lime production, thus leading to a higher profit for the company.

Page – 46

REUSE RESIDUAL SAND CASTING FOR PRODUCTION OF CONCRETE BLOCKS

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ABSTRACT

Sand casting is a viable waste as an alternative raw material, regarding a good solution in the production of concrete blocks. The use of sand casting to produce concrete blocks is performed by about 80% of the productive capacity of the company considered in this study. Thus, this work aims to make an improvement by applying fuzzy logic. The methodology was applied from the formulation of ranking criteria, which improved the production process. Therefore, after the implementation of the established criteria, was achieved an improvement of about 10%. Thus, the results achieved can help companies, generating alternatives with more excellent reliability and assertiveness. Moreover, Fuzzy is a methodology that provides strong support for decision making, supporting subjective and intuitive criteria.

Page – 55

E-SELECTIN AS A BIOMARKER IN FEMALE PATIENTS WITH B-THALASSEMIA IN AL- NAJAF PROVINCE, IRAQ

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ABSTRACT

E-selectin, as identified (CD62E), is expressed on endothelial cells after stimulation with inflammation cytokines. β -Thalassemia diseases (β T) and early diagnosis are of utmost significance in the entire world population. This study was performed in the Thalassemia Center of the Al-Zahraa Educational Hospital in Al-Najaf Province, Iraq, on sixty-nine with β -thalassemia (54 β T major and 15 β T Intermedia) aged 8-40 years who transfused blood. Compared to 20 healthy volunteers as a control group. In both β T patients and healthy groups were assessed serum E-selectin levels. It was investigated the relationship with RBC, Hb, PCV, WBC, PLT, BMI, splenic status, iron, and ferritin levels. The results revealed a significant ($P<0.05$) decreased values of HB, RBC, P.C.V, and BMI. In contrast, values of WBC, PLT, Iron, and Ferritin were significantly increased in β T patients as compared to the healthy control groups. A significant ($P<0.05$) increase in serum E- Selectin level in β T patients (20.55 ± 0.47) ng/ml to compare with the healthy group (9.16 ± 0.50) ng/ml. Furthermore, it was a significant decrease in groups of β T major (19.87 ± 0.42) ng/ml more than in β T intermedia (23 ± 1.42) ng/ml. E-Selectin revealed a significant increase ($P<0.05$) in progress age and associated with splenectomies and underweight groups compared to splenectomies and the normal weight groups, respectively. Also, E-Selectin levels significantly positively correlated with WBC, PLT value, iron, and Ferritin levels. However, it was no significant with RBC, PCV, Hb. As a conclusion from this study, E- Selectin is an important biomarker in β -thalassemia patients can be identified as the complications associated with iron overload, inflammatory process, and endothelial dysfunction in β T disease.

Page - 66

TO AUTHORS

Preparation of Manuscripts	74
Guidelines for publication	77
How to submit a manuscript?	78
Invitation	79

(Cu_{0.4}Al_{0.3})TaSe₂: PREPARATION AND CRYSTAL STRUCTURE ANALYSIS FROM X-RAY POWDER DIFFRACTION

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ABSTRACT

A new phase of the (CuAlSe₂)_{1-x}(TaSe)_x alloy system was synthesized by the melt and annealing technique and studied by SEM, DTA, and XRPD techniques. Its structure has been refined by the Rietveld method using X-ray powder diffraction data. The new alloy corresponds with the stoichiometry Cu_{0.4}Al_{0.3}TaSe₂. This compound crystallizes in the hexagonal space group $P\bar{6}m2$ (N° 187) with a MoS₂-type structure, and unit cell parameters $a = 3.455(2)$ Å, $c = 13.423(4)$ Å, $V = 138.7(1)$ Å³, $Z = 2$. The crystal structure is based on the MoS₂-type of stacking of TaSe₂ layers with a partial ordering of Cu and Al cations over the tetrahedral sites. The powder pattern was composed of 63.1% of the principal phase Cu_{0.4}Al_{0.3}TaSe₂ and 29.9% of CuAlSe₂, 7.0% of TaSe₃, as the secondary phases

Keywords: Alloys, X-ray diffraction, crystal structure, MoS₂ type

1. INTRODUCTION

The compounds with ternary structures of the chalcopyrite family Cu-III-VI₂ (III = Al, Ga, In, VI = S, Se, Te) form a wide group of semiconductor materials with diverse optical and electrical properties (Shay and Wernik, 1974). They crystallize with tetragonal symmetry, and the addition of an II-VI (II = Mn, Fe, Co, Ni, Ta) binary compound produces alloys of the type (Cu-III-VI₂)_{1-x}(II-VI)_x (Parthé, 1995). The formation of some member with compositions Cu₂-II-III-Se₅ ($x = \frac{1}{3}$), Cu-II-III-Se₃ ($x = \frac{1}{2}$), Cu-II₂-III-Se₄ ($x = \frac{2}{3}$) have been reported (Grima-Gallardo *et al.*, 2000;

2001a; 2001b; Mora *et al.*, 2007; Delgado *et al.*, 2008; 2019). All these phases fulfill the rules of formation of adamantane compounds and belong to the normal semiconductor compound families derivatives of the II-VI binary semiconductors (Parthé, 1995).

These type of semiconductors combined with magnetic behavior give place to the so-called "diluted magnetic semiconductors" or "semi-magnetic semiconductors", which have been largely investigated in the last years due to their possible application in spintronic devices (Jain, 1991; Pearton *et al.*, 2003). Besides, chalcogen-based (VI = S, Se, Te) begin to be studied as potential trainers of "wide bandgap

semiconductors", which are essential for electronic devices and energy applications because of their high optical transparency, controllable carrier concentration, and tunable electrical conductivity (Woods-Robinson *et al.*, 2020). On the other hand, Ta-based chalcogenides are usually used to form alloys with applicable properties due to their high melting point (Kikkawa *et al.*, 1982; Hayashi *et al.*, 2007; Ali *et al.*, 2015).

In this work, and as part of ongoing structural studies on semiconductors of the system $(\text{Cu-III-VI}_2)_{1-x}(\text{Ta-VI})_x$ (Grima-Gallardo *et al.*, 2007; 2008; 2018a; 2018b; Delgado *et al.*, 2007; 2008), we report the synthesis and structural analysis of the quaternary semiconductor with composition $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$. This compound is a new member of the $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ family, which is a related phase of the hexagonal compound $2\text{H-Cu}_{0.52}\text{TaSe}_2$ (Ali *et al.*, 2015).

2. MATERIALS AND METHODS

2.1. Preparation

Starting materials (Cu, Al, Ta, and Se) with nominal purity of 99.99 wt. % (Goodfellow) in the stoichiometric ratio were mixed in an evacuated (10^{-4} Torr) and sealed quartz tube with the inner walls previously carbonized to prevent the chemical reaction of the elements with quartz. The quartz ampoule is heated until 493 K (melting point of Se), keeping this temperature for 48 h and shaking all the time using an electromechanical motor. This procedure guarantees the formation of binary species at low temperatures avoiding the existence of Se free gas at high temperature, which could produce explosions or Se deficiency in the ingot. Then the temperature was slowly increased until 1423 K, with the mechanical shaker always connected for better mixing of the components. After 24 h, the cooling cycle begins until the anneal temperature (800K) with the mechanical shaker is disconnected. The ampoule is keeping at the annealing temperature for 1 month to ensure the thermal equilibrium. Then the furnace is switching off.

2.2. Scanning Electron Microscopy

Stoichiometric relations of the sample were investigated by scanning electron microscopy (SEM) technique, using a Hitachi S2500 equipment. The microchemical composition was found by an energy-dispersive x-ray spectrometer (EDS) coupled with a computer-based

multichannel analyzer (MCA, Delta III analysis, and Quantex software, Kevex). For the EDS analysis, $K\alpha$ lines were used. The accelerating voltage was 15 kV. The samples were tilted 35 degrees. A standardless EDS analysis was made with a relative error of ± 5 -10% and detection limits of the order of 0.3 wt %, where the k-ratios are based on theoretical standards.

2.3. Differential Thermal Analysis

Differential Thermal Analysis (DTA) measurements were carried out in a fully automatic Perkin-Elmer apparatus, which consists of a Khantal resistance furnace ($T_{\text{max}} = 1650$ K) equipped with Pt/Pt-Rh thermocouples and an informatics system for the automatic acquisition data. The internal standard used was a high purity (99.99 wt. %) piece of gold. The temperature runs have been performed from ambient temperature to 1400-1500 K, which is the recommended operative limit. The heating rate was controlled electronically to 20 Kh⁻¹; the cooling rate was given by the natural cooling of the furnace after switching off. From the thermogram, transition temperatures were manually obtained from the ΔT vs. T graph with the criteria that the transition occurs at the intersection of the baseline with the slope of the thermal transition peak, as usual. The maximum error committed in the determination of transition temperatures by this method was estimated to be ± 10 K.

2.4. X-Ray Powder Diffraction

X-ray powder diffraction data were collected employing a diffractometer (Siemens D5005) equipped with a graphite monochromator ($\text{CuK}\alpha$, $\lambda = 1.54059$ Å) at 40 kV and 20 mA. Quartz was used as an external standard. The sample was scanned from 5 - 100° 2θ , with a step size of 0.02° and counting time of 20 s. The peak positions were extracted utilizing single-peak profile fitting carried out through the Bruker Diffracplus software. For the Rietveld refinement, the whole diffraction data was used.

3. RESULTS AND DISCUSSION:

Three different regions of the ingot were scanned, and the average atomic percentages from SEM are summarized in Table 1.

Table 1. SEM experimental results for $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ alloy system for $x = \frac{1}{2}$

Nominal stoichiometry (%)	Experimental stoichiometry (%)
Cu=16.67	Cu = 09.8 ± 0.3
Al=16.67	Al = 07.0 ± 0.5
Ta=16.67	Ta = 29.8 ± 0.4
Se=50.00	Se = 53.4 ± 0.6

The mean experimental stoichiometry observed has been calculated as $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$. In Figure 1, the microphotography of this phase is shown. It can be observed the platelets that confirms the laminar character of the crystal structure for this phase.

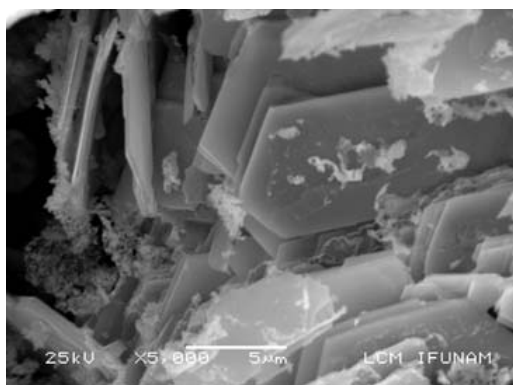


Figure 1. Microphotography of the hexagonal $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ alloy system for $x = \frac{1}{2}$

In Figure 2, DTA heating and cooling cycles for $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ $x = \frac{1}{2}$ alloy are plotted. In the heating cycle, the sample shows a peritectic melting point with a relatively wide solid+liquid region. The differential thermal analysis (DTA) indicates that this compound melts at 1445 K.

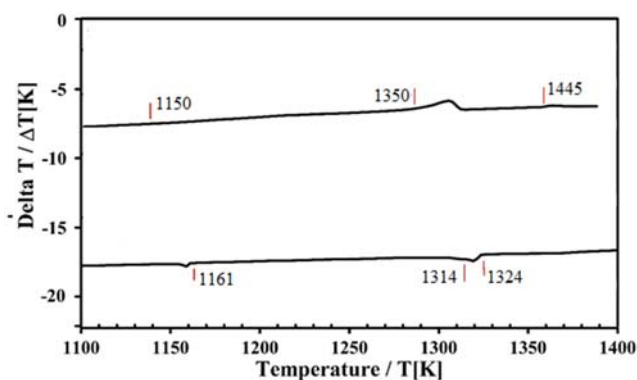


Figure 2. DTA heating (top) and cooling (bottom) cycles for $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ alloys with $x = \frac{1}{2}$

Figure 3 shows the resulting X-ray powder diffractogram. An automatic search in the PDF-ICDD database (ICDD, 2013) indicates that the powder pattern also contains small amounts of the ternary CuAlSe_2 (PDF 01-075-0101) and the binary TaSe_3 (PDF 00-18-1310). Bragg positions of the diffraction lines from these phases are also indicated in Figure 3. The remaining intense peaks corresponding to the phase of interest were indexed in a hexagonal cell with: $a = 3.454 \text{ \AA}$ and $c = 13.411 \text{ \AA}$, using the program Dicvol04 (Boultif and Louër, 2004). A revision of the diffraction lines of the main phase taking into account the sample composition and unit cell parameters suggested that this material is isostructural with the phase $2\text{H-Cu}_{0.52}\text{TaSe}_2$, recently reported, which crystallize in the space group $P\bar{6}m2$ (N° 187) (Ali *et al.*, 2015).

Structural refinement was performed with the Rietveld method (Rietveld, 1969) using the program Fullprof Suite (Rodríguez-Carvajal, 1993) by applying a Cox-Hastings pseudo-Voigt function (Thompson *et al.*, 1987). The background was fitted using a linear interpolation between a set of background points with refinable heights. The angular dependence of the peak full width at half maximum (FWHM) was described by the Caglioti's formula (Caglioti *et al.*, 1958). The thermal motion of the atoms was described by one overall isotropic temperature factor for each phase.

The atomic coordinates of $\text{Cu}_{0.52}\text{TaSe}_2$ (Ali *et al.*, 2015) were used as a starting model for the quaternary $(\text{CuAl})_x\text{TaSe}_2$, where the occupation factors of Cu and Al atoms were refined. The atomic positions of the ternary CuAlSe_2 (Hahn *et al.*, 1953) and the binary TaSe_3 (Bjerkelund and Kjekshus, 1965) were included as secondary phases in the refinement.

The results of the Rietveld refinement are summarized in Table 2. The final Rietveld refinement converged to the following weight fraction percentages: $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$ (63.1%), CuAlSe_2 (29.9%) and TaSe_3 (7.0%).

It should be noted that the occupancy factors for Cu and Al cations are in good agreement with the values found in chemical analysis (see Table 3), and the stoichiometry of this alloy is: $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$.

Figure 3 shows the observed, calculated, and different profiles for the final cycle of Rietveld refinement. Atomic coordinates and isotropic temperature factor for the new phase $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$ are shown in Table 3.

Table 2. Rietveld refinement results for $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$

Molecular formula	$\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$
Molecular weight (g/mol)	373.56
System	hexagonal
Space group	$P\bar{6}m2$ (187)
D_{calc} (g/cm ³)	2.22
a (Å)	3.455(2)
c (Å)	13.423(4)
V (Å ³)	138.7(1)
Z	2
R_{exp} (%)	7.8
R_p (%)	8.8
R_{wp} (%)	9.1
S	1.2
R_B (%)	8.5

$$R_p = 100 \sum |y_{\text{obs}} - y_{\text{calc}}| / \sum |y_{\text{obs}}| \quad S = [R_{\text{wp}} / R_{\text{exp}}]$$

$$R_{\text{exp}} = 100[(N+C)/\sum w(y_{\text{obs}}^2)]^{1/2} \quad R_B = 100 \sum_k |l_k - l_{\text{calc}}| / \sum_k |l_k|$$

$$R_{\text{wp}} = 100 [\sum w(y_{\text{obs}} - y_{\text{calc}})^2 / \sum w |y_{\text{obs}}|^2]^{1/2}$$

$N-P+C$ = degrees of freedom

The crystal structure is based on the MoS_2 type of stacking of TaSe_2 layers with a partial ordering of Cu and Al cations over the tetrahedral sites. Figure 4 shows the tetrahedral around the Cu and Al cations with the TaSe_2 layer at the center.

$\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$ is a new chalcogenide compound related to the layered $2\text{H-Cu}_{0.52}\text{TaSe}_2$ phase, with potential use as a wide bandgap semiconductor (Woods-Robinson *et al.*, 2020).

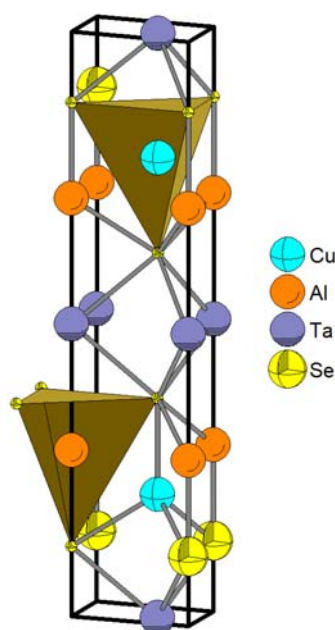


Figure 4. Unit cell representation for $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$, showing the tetrahedral around the Cu and Al cations

4. CONCLUSIONS:

The phase with $x = 1/2$ for the alloy system $(\text{CuAlSe}_2)_{1-x}(\text{TaSe})_x$ has been synthesized and studied by SEM, DTA, and XRPD techniques. A new phase with experimental stoichiometry $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$ was identified and refined. This alloy crystallizes in the hexagonal structure, space group $P\bar{6}m2$ (N° 187), with unit cell parameters $a = 3.455(2)$ Å, $c = 13.423(4)$ Å, $V = 138.7(1)$ Å³. The powder pattern was composed of 63.1% of the principal phase $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$ and 29.9% of CuAlSe_2 , 7.0% of TaSe_3 , as the secondary phases.

5. ACKNOWLEDGMENTS

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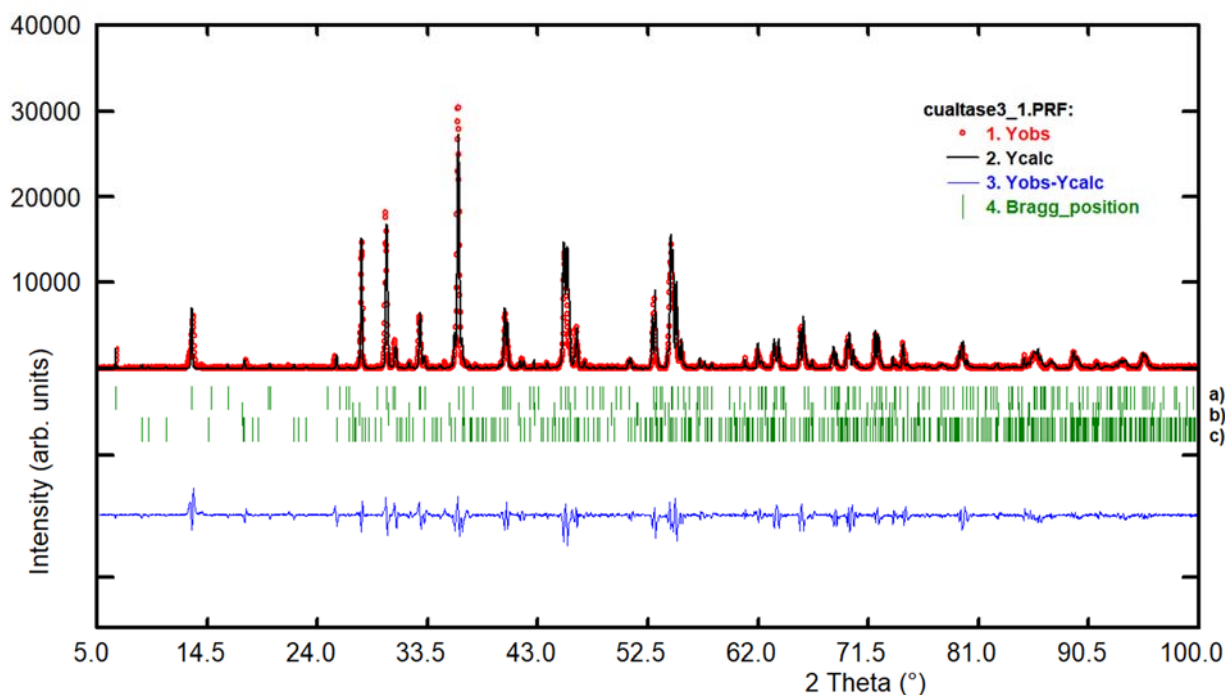


Figure 3. Rietveld refinement of the powder pattern shown the new phase a) $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$, together with the secondary phases b) CuAlSe_2 and c) TaSe_3 . The lower trace is the difference curve between observed and calculated patterns. The Bragg reflections are indicated by vertical bars

Table 3. Atomic coordinates (\AA) and isotropic temperature factors for $\text{Cu}_{0.4}\text{Al}_{0.3}\text{TaSe}_2$, derived from the Rietveld refinement.

Atom	Ox.	Site	x	y	z	foc	B (\AA^2)
Cu	+1	2i	$\frac{2}{3}$	$\frac{1}{3}$	0.2124(8)	0.41(2)	0.6(4)
Al	+3	2g	0	0	0.2845(7)	0.32(2)	0.6(4)
Ta1	+2	1b	0	0	$\frac{1}{2}$	1	0.6(4)
Ta2	+2	1e	$\frac{2}{3}$	$\frac{1}{3}$	0	1	0.6(4)
Se1	-2	2i	$\frac{2}{3}$	$\frac{1}{3}$	0.3762(7)	1	0.6(4)
Se2	-2	2g	0	0	0.1205(7)	1	0.6(4)

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ABSTRACT

Diabetes mellitus (DM) is a clinical disease correlated with a deficiency of insulin secretion or action. It is one of the leading causes of morbidity and mortality worldwide. The global burden of diabetes is rising due to increasing obesity and population aging. Urinary tract infections (UTI) are common microbial infections known to affect the different parts of the urinary tract accounting for major antibacterial drug consumption. About 150 million UTI cases were diagnosed every year. Urinary tract infections are the most important and most common site of infections in a diabetic patient. Diabetic patients have been found to have a 5-fold frequency of acute pyelonephritis at autopsy than non-diabetics. Most of the urinary tract infections in patients with diabetes are relatively asymptomatic. The presence of this syndrome predisposes to much more severe infections, particularly in patients with acute ketoacidosis, poor diabetic control, diabetic complications such as neuropathy, vasculopathy, and nephropathy. The Gram-negative aerobic bacilli are the large group of bacterial pathogens that cause UTI with few species of Gram-positive bacteria. However, some fungi, parasites, and viruses have also been reported to invade the urinary tract. Urinary tract infection affects women more than men due to several factors such as proximity of the genital tract to the urethra, anatomy of the female urethra, sexual activity, menopause, and pregnancy. Other possible risk factors of UTI include allergy, obesity, diabetes, past history of UTI, contraceptive use, catheter use, and family history.

Keywords: *Diabetes mellitus, urinary tract infections, risk factors, pathogenesis, antibiotic resistance.*

1. INTRODUCTION:

Diabetes mellitus is related to immune system dysfunction, which makes the afflicted individuals prone to frequent infections, especially infections of the genital and urinary tract. Diabetic patients have more than twice the opportunity of developing genitourinary tract infections (Abu-Ashour *et al.*, 2017). Few researches have shown that the cause could be due to dysfunctional bladders contracting poorly may make static pools of urine that serve as favorable media for microbial growth. Other studies suggest that urine of hyperglycemic patients encourages increased bacterial load and colonization in the urinary tract (Njunda *et al.*, 2013). These and other causes make the genitourinary system where UTI can be a cause of severe life-threatening complications such as emphysematous pyelonephritis, emphysematous cystitis, and renal papillary necrosis, which are common in a diabetic patient that leads to kidney failure in these patients (Casqueiro *et al.*, 2012).

The bacterial strains, mostly *E. coli*, can cause UTI in both females and males, while *Staphylococcus saprophyticus* have been found to cause UTI mostly in younger females and can also occur secondary to blood-borne infections (Anuja and Shah 2015). Others include *Pseudomonas aeruginosa*, *Klebsiella spp*, *Proteus mirabilis*, *Morganella spp*, and *Enterobacter spp*. Also implicated are *Enterococcus faecalis* and *Staphylococcus aureus* (Rubin *et al.*, 1992). Urinary tract infections in older males and females with the indwelling catheter are mostly occurring as a result of *Proteus spp* and *P. aeruginosa* (Cheesbrough M. 2010). These bacteria that cause UTI initially proliferate at the opening of the urethra and ascend to the bladder, while some may reach to the kidneys from the bloodstream (Gupta *et al.*, 2011). Infections of fungi are common among patients with exhausting diseases and structural abnormalities of the urinary tract, which are infrequent causes of UTIs in healthy persons (Fisher *et al.*, 2011). However, bacterial pathogens are the highest cause for the

majority of UTI incident in diabetic patients, determination of *Candida* spp. in urine cause a diagnostic challenge (Fisher, JF 2011).

It is very important to investigate diabetic patients for UTI for suitable diagnosis, whole treatment, and avoidance of progression to kidney complications and, finally, severe renal failure. However, there are controversies regarding the clinical pattern, incidence, and microbiology of UTIs in diabetic persons as compared to those non-diabetic ones (Aswani *et al.*, 2014). The aim of this study was to study the correlated between UTIs and diabetes mellitus, study many important factors that may play an achievable role in the incidence the UTIs in diabetic patients. Hence, this study would help assess the frequency of UTIs in the diabetic population and enable the diabetologists to predict the clinical and microbiological patterns of UTI in their patients.

1.1 Urinary Tract Infection in Diabetic Women

The larger cases of UTI were found in women compared to men both in diabetic and non-diabetic individuals. The majority of the study done all over the world has concluded female predominance to UTI over (Geerlings *et al.*, 2008). The higher rate of occurrence UTIs among female patients is due to the short urethra and its proximity to the anus (Chhetri *et al.*, 2001), pregnancy, sexual activity, menopause (Schaeffer *et al.*, 2001), perineal contamination of the urinary tract with fecal micro-flora, and the absence of prostatic secretion (Pargavi *et al.*, 2011). According to a study that achieved at the University of Uyo Teaching Hospital in Nigeria, The majority of outpatients presenting with symptoms and signs of urinary tract infections in Uyo do not have Microbiological confirmed UTI as only about 30% of women with presenting signs of UTI had positive results for urine culture tests while only about 12% of men with these sign are culture positive for UTI (Abraham *et al.*, 2019). Urinary bladder dysfunction occurs in 26%–85% of diabetic females, depending on the duration of diabetic disease and the age extent of neuropathy, and thus should be considered in all diabetic patients with infections of the urinary tract (Frimodt-Møller C. 1980).

1.2 Pathogenesis

Several potential mechanisms unique to diabetes may contribute to the increased risk of UTI in patients with diabetes (Chen *et al.*, 2009).

Higher concentrations of glucose in the urine may promote the growth of harmful bacteria (Wang *et al.*, 2013). However, several studies did not find a relationship between HbA1c levels, which serves as a risk of UTI among diabetic patients, and a proxy for glycosuria; also, sodium-glucose cotransporter two inhibitors, which increase glycosuria, were not found to increase the rate of urinary tract infections (Boyko *et al.*, 2002). High renal parenchymal glucose levels make a favorable environment for the growth and proliferation of microorganisms, which might be one of the precipitating factors of pyelonephritis and the complications such as emphysematous pyelonephritis (Soo Park *et al.*, 2006). Various impairments in the immune system, including innate, humoral, and cellular immunity, may contribute to the pathogenesis of UTI in patients with diabetes (Geerlings SE *et al.*, 2000).

1.3 Risk factors

Asymptomatic Bacteriuria (ASB) is more predominant in women due to a short urethra that is in proximity to the moist, warm, vulvar, and perianal areas that are colonized with intestinal bacteria. ASB increases with age and is also associated with foreign bodies or urinary tract abnormalities (Colgan *et al.*, 2006; Nicolle LE 2014). Numerous studies have reported an increased incidence of ASB in diabetic patients, with estimations ranging from 8%–26% (Zhanel *et al.*, 1995). A meta-analysis of 22 studies, issued in 2011, found a point frequency of 12.2% of ASB among diabetic patients versus 4.5% in healthy control individuals (Schneeberger *et al.*, 2014). The point prevalence of ASB was higher in patients with a longer duration of diabetes, was higher both in men and women and was not correlated with glycemic status, as estimated by glycosylated hemoglobin A1c (HbA1c) (Renko *et al.*, 2011). A current prospective study of inpatients at an Indian hospital found a 30% prevalence rate of asymptomatic bacteriuria among diabetic patients (Aswani *et al.*, 2014).

Pyelonephritis was found to be 4.1 times more recurrent in pre-menopausal diabetic women than in non-diabetic women in a case-control study of a Washington State health group (Scholes *et al.*, 2005). In a Canadian study, diabetic women (type 2 and 1, identified by receipt of oral hypoglycemic or insulin therapy) were 6–15 times more commonly hospitalized (according to age group) for acute pyelonephritis than non-diabetic women, and also diabetic men

were hospitalized 3.4–17 times more than non-diabetic men (Nicolle *et al.*, 1996). A study of Danish detected that patients with diabetes mellitus were three times more likely to be hospitalized with pyelonephritis, as compared to subjects without diabetes (Benfield *et al.*, 2007).

1.4 The Pathogens and Antibiotic Resistance

Escherichia coli (*E. coli*) are the most common pathogens isolated from the urine of diabetic patients with UTI, other Enterobacteriaceae such as *Proteus* spp., *Klebsiella* spp., *Enterobacter* spp., and Enterococci (Geerlings *et al.*, 2002). *Escherichia coli* is the notable causative pathogens of UTI in both diabetic and non-diabetic people, followed by coagulase-negative *Staphylococci* (CONS), *Enterococcus* species (spp.), *Candida albicans*, and non-albicans *Candida* spp (Woldemariam *et al.*, 2019; Bollestad *et al.*, 2018). *Staphylococcus aureus* also constitutes the most pathogens responsible for UTI in diabetic patients, since those patients are classified as immunocompromised, and the bacteria *S. aureus* are opportunists. Moreover; the several virulence factors featured by *S. aureus* they have the ability to resist the most common antibiotics used to treat UTI as the drug of choice, pointing to the beta-lactam group of antibacterial antibiotics, they usually named Methicillin-resistant *S. aureus* (MRSA) (Martin *et al.*, 2014; Budiman *et al.*, 2018). Patients with diabetes are more prone to have resistant pathogens as the cause of their UTI, comprising fluoroquinolone-resistant uropathogens (Wu *et al.*, 2014), extended-spectrum β -lactamase-positive Enterobacteriaceae (Inns *et al.*, 2014; Colodner *et al.*, 2004), vancomycin-resistant Enterococci (Papadimitriou-Olivgeris *et al.*, 2014), and carbapenem-resistant Enterobacteriaceae (Schechner *et al.*, 2013). This might be due to many factors, including numerous courses of antibiotic therapy that are administered to these patients, frequently for asymptomatic or only mildly symptomatic UTI, and catheter-associated UTI and increased incidence of hospital-acquired, which are both associated with resistant pathogens. Type 2 diabetes is also a predisposing factor for fungal urinary infection (Sobel *et al.*, 2011).

2. MATERIALS AND METHODS

Approximately all studies in this review were used the same materials and methods for determining diabetes mellitus and UTIs in the selective persons of each study.

2.1 The Collection of samples

Almost in all studies, blood samples were collected from the patients visiting the hospital or any health location to select the diabetic patients included in this researches. Then, urine samples were collected as an aseptic technique as possible in a sterile universal tube. The collected samples were transported to the laboratories within 30 minutes of collection. If they could not, then the urine specimens were stored at 4 °C to prevent bacterial growth in the urine.

2.2 Laboratory Analysis

2.2.1 Macroscopic Examination

Tektook *et al.* (2017) achieved routine urinalysis for each sample to determine the turbidity, color; specific gravity; reaction; Sugar and Ketone bodies as well as Albumin.

2.2.2 Microscopic Examination

Depending on a cross-sectional study carried out in Uyo Teaching Hospital by Abraham *et al.* (2019), Ten millimeters of midstream urine was centrifuged for 5 minutes at 1,500rpm. The urine deposits were placed on a glass slide, covered with a coverslip, and examined by using 10x and 40x objectives to quantify the number of red blood cells, white blood cells, epithelial cells, calcium oxalate crystals, and cast present for possible detection of pyuria or bacteriuria. Gram stain was used to differentiate Gram-negative uropathogens from Gram-positive ones.

2.2.3 Bacterial Isolation, Culture, and Colony Counts

According to a hospital laboratory-based cross-sectional study of (Narayani *et al.*, 2018), bacteria were isolated from urine samples and cultured on Mac-Conkey agar and blood agar by the Semi-Quantitative method. Sample with more than 10⁵ colony-forming units (CFU)/mL bacteria were considered as positive. Isolation and identification of the microorganisms were done following standard laboratory protocol as per the American Society of Microbiology (ASM) (Isenberg, 2002). Antibiotic sensitivity tests of isolates were tested by the Kirby-Bauer disc diffusion method. The antibiotic discs used were ciprofloxacin, amoxicillin, ceftazidime, cotrimoxazole, cefotaxime, cefixime, gentamicin, cephalixin, ofloxacin, vancomycin, and nitrofurantoin. Results were read according to

Clinical Laboratory Standards Institute (CLSI) guidelines (2014).

In a prospective cross-sectional study of (Borowczyk *et al.*, 2017), antimicrobial sensitivity (susceptibility) or resistance also was performed by the Kirby–Bauer disk diffusion susceptibility test while it was calculated in accordance with the European Committee on Antimicrobial Susceptibility Testing (EUCAST) criteria (2014).

3. RESULT AND DISCUSSION:

3.1 RESULT

The higher cases of UTIs were found in females compared to males both in non-diabetic and diabetic patients. The majority of the studies achieved all over the world have concluded females predominance to UTIs over males (Akbar, 2001; Bonadio *et al.*, 2006; Boroumand *et al.*, 2006 and Geerlings, 2008). According to Table 1, which belongs to the study of (Abraham *et al.*, 2019), we also observed that the prevalence of UTI was higher in females 37 (25.9%) than in males 26 (11.5%) with a highly significant P-value (0.0001). In table 2, which demonstrated the association of certain risk factors with pyelonephritis, we found that the study of (Scholes *et al.*, 2005) considered the diabetes mellitus as a risk factor for pyelonephritis as well as other factors such as UTIs, chlamydial infection, sexually transmitted diseases (STD), sexual intercourse, hypertension, and Any antibiotic use. The majority of studies concluded that the Gram-negative bacteria were highly predominant in diabetic people with UTIs when compared to Gram-positive bacteria. Furthermore, *E. coli* and *Klebsiella* species were the most prevalent bacterial infections in those groups of people (Kolawole *et al.*, 2009; Inabo *et al.*, 2006; Kehinde *et al.*, 2011; Abraham *et al.*, 2019). Also, another study which was achieved in Iraq showed that *E. coli* and *Klebsiella pneumoniae* were the major pathogens in diabetic patients (table 3) (Tektook *et al.*, 2017). According to the study of (Narayani *et al.*, 2018), the antibiotic susceptibility pattern of *E. coli* and *K. pneumoniae* was showed in Table 4 as an example of the resistance of bacteria in diabetic patients. Moreover, they found that all *K. pneumoniae* isolates were multidrug-resistant (MDR) in diabetic, whereas 66.6% were MDR in non-diabetic patients. Likewise, *S. aureus* showed 100% MDR in diabetic patients, whereas in non-diabetic patients, 75% were found as MDR strain. There are several studies were had similar results in

case of bacterial resistance such as ((Maharjan *et al.*, 2015; Puri *et al.*, 2006 and Jha and Bapat, 2005; Borowczyk *et al.*, 2017).

3.2 DISCUSSIONS

Urinary tract infections (UTI) are common bacterial infections known to affect the different parts of the urinary tract accounting for large antimicrobial drug consumption (Dias Neto *et al.*, 2003). About 150 million UTI cases were identified every year (Akram *et al.*, 2007). UTIs are mainly defined as the colonization of a varied population of microorganisms colonizing in the urinary tract. From a microbiological viewpoint, UTI can occur anywhere, including the kidneys, bladder, ureters, and urethra (Hackett G 2005).

The larger susceptibility to infection in diabetic patient is due to the hyperglycemic environment that favors immune dysfunction (the reduced response of T cells, damage to the neutrophil function, humoral immunity, depression of the antioxidant system), gastrointestinal and urinary dysmotility, micro- and macroangiopathies, neuropathy, decrease in the antibacterial activity of urine, and the greater number of medical interventions required in these patients (Casqueiro and Alves 2012; Muller *et al.*, 2005). The Gram-negative bacilli, a class of bacteria, were highly prevailing in the UTI among diabetic patients when compared to Gram-positive bacteria, particularly cocci. Among the Gram-negative bacterial species, *Escherichia coli* were the most predominant uropathogenic, followed by *Klebsiella pneumoniae*. *Staphylococcus aureus* was the most prevalent Gram-positive cocci (Oluremi *et al.*, 2011).

Antibiotic is the cornerstone for treating bacterial infection. Emergence in resistance of bacteria against antibiotics are the main barrier against infection. According to a current study established in China, The effective drug against *S. aureus* was found to be vancomycin (susceptibility of 50%) and gentamicin (susceptibility of 50%) in diabetic patients, while in non-diabetic patients, the most effective drugs found to be gentamicin (susceptibility of 100%) followed by ofloxacin, ciprofloxacin, and vancomycin, (75.0 % each). *S. aureus* was highly resistant to cephalixin, amoxicillin, cotrimoxazole, ofloxacin, and ciprofloxacin (100% each) followed by ceftazidime (50.0%) in diabetic patients. In the non-diabetic group also, it was highly resistant to amoxicillin (75.0%) followed by ceftazidime and cotrimoxazole (50.0 %)(Narayani *et al.*, 2018).

Antibiotic resistance is a large global health problem both for community and hospital-acquired infections (WHO 2011). This problem is challenging in low-income countries because of the irrational uses of antibiotics, high prevalence of infection, poor infection prevention practices, and over-the-counter availability of antibiotics. Hence the emerging occurrence of antibiotic resistance (Alemu *et al.*, 2012; Abera *et al.*, 2014) and DM in Ethiopia is a reason for concern for health care providers. According to the Iraqi study which shown that UTIs are higher among type II diabetic patients (81%) rather than type I (19%); this is correlated with the differences in the aetio-pathogenesis of each; since type I is an autoimmune disease characterized by dysregulation of the immune system and elevation of cellular infiltration at the β -cells of Langerhans cells in addition to autoantibodies, while type II is a metabolic syndrome characterized by miss control of glucose which may facilitate the bacterial growth (Wolde Gebre M 2013).

4. CONCLUSIONS:

Based on the mentioned studies, it was concluded that diabetic patients are at high risk of infections. The most frequent infection is urinary tract infection, affecting mostly the women, the most common causative organism being *E.coli*. Therefore, Stepping-up the prevention and early detection of UTIs in this group of women seems to be the best way to avoid future complications as well as performing urine culture, and constant surveillance of UTI on DM patients is necessary.

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Table 1. Prevalence of UTI in correlated with gender

Gender	No. of Examined samples	No. of Samples with positive culture (%)	Odds Ratio	P-value
Female	143	37 (25.9)	2.7	0.0001*
Male	227	26 (11.5)	1	
Total	370	63 (17.0)	-	

* Statistically significant, (χ^2 (1)=12.91; P=0.0001; OR=2.7)

Table 2. Association of certain risk factors with pyelonephritis

Variable	Case group, % (n = 242)	Control group, % (n = 546)	Odds Ratio	P-value
Diabetes	6.6	1.6	4.2 (1.8–9.7)	<0.001
Chlamydial infection	12.8	7.9	1.7 (1.0–2.8)	0.03
Other STD	28.1	30.8	0.9 (0.6–1.2)	>0.2
Any previous UTI	69.4	49.7	2.3 (1.7–3.2)	<0.001
Ever had sexual intercourse	98.8	91.2	7.6 (2.4–24.7)	<0.001
Hypertension	9.9	6.4	1.6 (0.9–2.8)	0.09
Any antibiotic use in the previous 30 day	15.8	8.1	2.1 (1.3–3.4)	<0.01

Table 3. Bacterial types causing UTIs in Diabetic patients

Type of Bacteria	No. of Isolates (%)	Type of D.M	
		I	II
<i>Escherichia coli</i>	15 (28,5)	2	13
<i>Klebsiella pneumoniae</i>	13 (24,5)	1	12
<i>Proteus mirabilis</i>	9 (17)	2	7
<i>Streptococcus agalactiae</i>	7 (13)	2	5
<i>Pseudomonas aeruginosa</i>	6 (11)	2	4
<i>Staphylococcus aureus</i>	3 (6)	1	2
Total (%)	53 (100)	10 (19)	43(81)

Table 4. Antibiotic Resistance of *E. coli* and *K. pneumoniae*

Antibiotic	Resistance of <i>E. coli</i>		Resistance of <i>K. pneumoniae</i>	
	Diabetic No. (%)	Non- diabetic No. (%)	Diabetic No. (%)	Non- diabetic No. (%)
Amoxicillin	81 (81.8)	64 (82.0)	3 (100)	5 (83.3)
Cefotaxime	39 (39.3)	23 (29.4)	0	2 (33.3)
Cefixime	36 (36.3)	26 (33.3)	0	2 (33.3)
Cotrimoxazole	33 (33.3)	32 (41.0)	0	3 (50.0)
Ciprofloxacin	42 (42.2)	29 (37.1)	3 (100)	1 (16.6)
Ofloxacin	48 (48.4)	30 (38.4)	3 (100)	2 (33.3)
Nitrofurantoin	3 (3.0)	9 (11.5)	0	2 (33.3)
Gentamicin	6 (6.0)	7 (8.9)	3 (100)	2 (33.3)

INVESTIGATING THE EFFECT OF COLD TEMPERATURE STRESS ON UNOPENED MALE CATKINS AND INOCULATED FEMALE FLOWERS OF IRANIAN NATIVE HAZELNUT CULTIVARS

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ABSTRACT

In many low-temperature areas, the environmental factor is an important limiting factor for the production and distribution of horticultural plants. This study aimed to investigate the cold tolerance of the male catkins and inoculated female flowers to screen the popular native hazelnut cultivars in Qazvin under low-temperature stress. A completely randomized factorial block design with three replications was used in this experiment with eight cultivars (Nakhnroud, Khandan, Mish-Pestan, South of Qarabagh, Asl-e-Qarabagh, Rasmi, and Gerdashkevar). After removing each of the treated samples at the end of the experiment, the samples were examined morphologically (appearance) and compared with the control. The changes were recorded as qualitative traits. To understand the influence of cold stress on reproductive organs, hydrogen peroxide and proline were measured. The results showed the onset of freezing in unopened male catkins at -7 and -9 °C and in inoculated female flowers at -3 °C. Damage to unopened male catkins' tissue occurred at -11 °C and in female flowers at -5 °C. The highest value observed among cultivars in the case for proline content of male catkins was in Mish-Pestan and Khandan cultivars with 0.816 and 0.660 $\mu\text{mol/g FW}$, respectively. In inoculated female flowers, Mish-Pestan and Tabestaneh cultivars with 0.185 and 0.168 $\mu\text{mol/g FW}$, respectively, showed the highest statistically significant increase in proline content. Interestingly, the cultivars with the highest proline content in male catkins indicated the most increase in H_2O_2 ; Mish-Pestan and Khnadan with 0.569 and 0.541 ug/g FW , respectively. Asl-e-Qarabagh was observed to have the least H_2O_2 content (0.042 ug/g FW) among cultivars. Again, in inoculated female flowers, those with the highest concentration of proline (Mish-Pestan and Tabestaneh) were found to have the highest H_2O_2 content (0.335 and 0.331 ug/g FW , respectively).

Keywords: Low-temperature stress, Proline, Hydrogen peroxide, Discoloration, Morphological traits

1. INTRODUCTION:

Plants face various environmental stresses during their lifetime, limiting the chances of plants growing and surviving. In many parts of the world, good growing conditions last only for a short time. In some places where the growing conditions are suitable, increasing the density and number of plants is a factor in creating competition for plants to obtain water and nutrients (Beck *et al.*, 2004; Peng *et al.*, 1996). Low temperature is the most important factor determining the distribution of plant species on the earth. And it can limit the yield and distribution of crops and orchards. Today, many plants are not native to their cultivated areas or derived from a cross between species or cultivars that are not native to the area. The importance of low temperatures in horticulture has been known since the beginning of agriculture; for example, the Romans selected species to be

cultivated in certain areas with cold weather (An *et al.*, 2019; Peng *et al.*, 1996).

Low temperatures in winter and the risk of spring frosts as important factors which nearly two thousand years ago, in the first century AD, efforts were made to protect crops from frost damage, but despite the various methods of frost protection that have been invented over time, frost damage to plants has been a major issue and a problem of great economic importance. This problem exists even in the subtropical regions (Molnar *et al.*, 2017).

Frost damage reduction has been devoted to addressing this important problem, not only through the development of conservation methods or delayed flowering but also through the study of physiological mechanisms involved in frost stress. However, this remarkable effort has only led to a relatively small improvement. The ultimate

reasons determining how plants can withstand the cold stress are still unclear to a large degree (Cristofori *et al.*, 2017; Kosenko *et al.*, 2019; Wanjiku and Bohne, 2015).

Therefore, frost is still one of the biggest causes of loss of agricultural products among all environmental and biological harmful factors (Saielli *et al.*, 2012; Sajadian *et al.*, 2019). Hazelnut is one of the nut trees that is of great economic value. The plant is mostly shrubby and is rarely seen as a tree. The main hazelnut growing areas around lakes and seas have mild winters and cool summers (Gönenc *et al.*, 2006; Linaldeddu *et al.*, 2016). The three major hazelnut growing areas in the world include Turkey (under the influence of the Black Sea), Italy, and Spain (Affected by the Pacific) (Saydut *et al.*, 2016; Silvestri *et al.*, 2020). The world production of hazelnuts in 2019 is estimated at 1037,500 tons. With a production of 780,000 tons, Turkey was the top producer of hazelnuts and produced 60% of the world's hazelnuts. Italy has 130,000 tons and the United States, with 30,000 tons, Spain with 25,000 tons.

In comparison, Iran has a production quantity of 18000 tons per year harvested from 20459 thousand hectares (ÇetİN *et al.* The yield of hazelnuts in Iran is very low and up to one ton per hectare, while in the major producer countries, it reaches 4 to 4.5 tons per hectare (Guliyev *et al.*, 2019; Mostashari-Rad *et al.*, 2020). Cold and frost are natural phenomena that cause a lot of damage to orchards, including hazelnuts, in some years. Cold damage to deciduous trees varies depending on the species and cultivar, and this action is often due to early spring frosts. Decreasing the temperature from the minimum tolerance threshold of the plant can be harmful. Gardeners are fully aware of the detrimental consequences of the trees' lack of resistance to winter cold (Çetinbaş-Genç *et al.*, 2020; Kosenko *et al.*, 2019; Silvestri *et al.*, 2020).

Thus, understanding how cold stress and frost occurs and its symptoms in each area by examining the hazelnuts trees in the area can guide and help agricultural planners and gardeners reduce frost damage. Additionally, screening native hazelnut cultivars to find the most resistance ones is a significant step in assisting the farmers to cultivate the cold stress tolerance cultivars to minimize the economic consequence of frost damage. The two outcomes mentioned above are the main reasons for carrying out this study.

2. MATERIALS AND METHODS:

2.1. Plant materials and treatments

To perform this experiment, 20-year-old hazelnut trees are located at the Agricultural and Natural Resources Research Center of Qazvin Province (East of Alamut). Factors in this experiment were: first, native hazelnut cultivars (Nakhnroud, Khandan, Mish-Pestan, South of Qarabagh, Asl-e-Qarabagh, Rasmi, and Gerdashkevar), the second: cold intensity in ten temperature levels (+3, +1, -1, -3, -5, -7, -9, -11, -13, -15 °C), using an incubator and the third; the cooling time was one hour in the flowering stage (unopened male catkins and female flower inoculated). Samples prepared from each cultivar were placed in the incubator for one hour at each mentioned temperature. After applying cold treatments, the samples were taken out of the incubator, and the extent of damage to each sample was examined (Figure 1).

2.2. Morphological traits

Morphology, including (superficial tissue discoloration, inner tissue discoloration, tissue margin burn, weak freezing, complete tissue freezing, tissue loss after 24 hours), were examined. Then the changes were performed as a qualitative scoring table at level five. Score: zero (no change), one (low change), three (medium change), five (high change), seven (very high change), each of which includes a series of morphological changes at each level.

2.3. Determination Hydrogen peroxide

The H₂O₂ content was estimated via Çatav *et al.* (2020) method. Fresh tissue (0.3 g) was homogenized in 0.1% TCA and centrifuged at 12000 rpm for 15 min. The supernatant (0.5 mL) was added to 0.5 ml potassium phosphate buffer (pH 7.0), and 1 ml potassium iodide (1 M) and absorbance were recorded by spectrophotometer at 390 nm.

2.4. Determination of Proline

Proline content was measured, according to Rathika *et al.* (2020). Fresh material was homogenized in 5 mL sulfosalicylic acid (3%) and then was centrifuged at 13000 rpm for 20 min. Two mL of supernatant was mixed with acid ninhydrin (2 mL) and glacial acetic acid (2 mL) and then was boiled at 100°C for one hour. The reaction mixture was extracted with 4 mL toluene, and the absorbance was recorded at 520 nm.

2.5. Statistical analysis

The statistical design of the factorial experiment in a completely randomized block design with three replications (two samples in each experimental unit) was used. SAS software was used for statistical analysis of data. A comparison of the means was performed by Duncan test at the level of 1% and 5% probability. EXCEL software was used to draw the graphs.

3. RESULTS AND DISCUSSION:

Examination of the morphology of unopened catkins (Figure 2) showed that at 5 °C, the male catkins had shown sensitivity to cold stress. In unopened male catkins, the onset of freezing is often at -5 and -7 °C, while complete freezing and tissue loss occurs at -11 °C. Among the cultivars studied for unopened male catkins, Mish-Pestan with a freezing point at -9 °C and complete tissue destruction at -11 °C showed more resistance than other cultivars. The morphology study results of inoculated female flowers (Table 2) showed that the onset of freezing occurred at -3 °C, and complete freezing occurred at -5 and -7 °C, which among the studied cultivars, Tabestaneh showed the highest tolerance.

The comparison of the mean data in unopened male catkins and inoculated female flowers have been presented in Figures 2 and 3, respectively, indicated that with time and increasing cold intensity, the concentration of hydrogen peroxide and proline increased in most cultivars. However, the level of hydrogen peroxide did not enhance due to cold stress as much as the protective amino acid proline. In the unopened male catkins and inoculated female flowers, respectively, proline has reached its maximum at the temperature where the serious injury took place. That is, over time and with increasing cold intensity, the proline has increased further. The highest value observed among cultivars in the case for proline content of male catkins was in Mish-Pestan and Khandan cultivar with 0.816 and 0.660 $\mu\text{mol/g FW}$, respectively, however proline content of Tabestaneh cultivar (0.562) did not show a significant difference at 5% level of Duncan's test with Khandan.

Proline content in the Asl-e-Qarabagh cultivar was found to be the lowest (0.336 $\mu\text{mol/g FW}$) compared to other cultivars (Figure 2). In inoculated female flowers, Mish-Pestan and Tabestaneh cultivars with 0.185 and 0.168 $\mu\text{mol/g FW}$, respectively, showed the highest statistically significant increase in proline content

when exposed to cold stress, whereas Rasmi cultivar with 0.077 $\mu\text{mol/g FW}$ showed the least proline content (Figure 3). The accumulation of this amino acid in perennial plants from mid-autumn to mid-winter is a natural physiological event in nitrogen storage metabolism. It was reported that pistachio cultivars, Akbari and Ahmad Aghaei under cold stress, to maintain the water potential of their tissues increasing proline was a significant approach (Sajadian *et al.*, 2019), similar results were observed in our study.

Barand *et al.* (2020) showed that the proline content of leaves and fruits of pistachios cultivars exposed to cold stress significantly increased compared to the control. The amount of free proline in many plants in response to environmental stresses such as cold stress and drought increases to a large extent and stabilizes the membrane (Mansour and Salama, 2020). Research on winter wheat has also revealed a positive correlation between an increase in cold stress intensity and proline content, and often, resistant cultivars observed to have more proline than susceptible ones (Ignatenko *et al.*, 2019; Pál *et al.*, 2018; Venzhik *et al.*, 2016). This case has also been seen in citrus so that the rate of increase proline in Trifoliate orange leaves, known as a cold-resistant plant, was about 4 times higher than that of found in rough lemon leaves after cold treatment (Liu *et al.*, 2017; Mohammadrezakhani *et al.*, 2019).

The proline content in soybean increased by a decrease in temperature, and the maximum amount was in winter (Yadegari *et al.*, 2007; Yildiztugay *et al.*, 2017). In cold-sensitive plants, the increase in cellular proline is insufficient to increase resistance unless high proline levels occur before stress. (Savouré *et al.*, 1997). It can be concluded that proline in the flowering stage compared to its previous periods (from defoliation to the beginning of growth in buds) experienced a significant upward trend due to the conversion of proline from the storage phase to non-storage form and also other forms, especially the consumable form for the plant (Charest *et al.*, 1990) which is consistent with our research. The concentration of sugar and proline increases during cold resistance, while starch concentration decreases (Patton *et al.*, 2007).

As shown in Figures 4 and 5, in the unopened male catkins and the inoculated female flower, respectively, the enzyme hydrogen peroxide levels increased in some cultivars and decreased in others. This research indicated that the excessive increase of hydrogen peroxide increases free radicals and active single oxygen

species, which in turn impose significant damage. Interestingly, the cultivars with the highest proline content in male catkins indicated the most increase in H_2O_2 ; Mish-Pestan and Khnadan with 0.569 and 0.541 $\mu\text{g/g}$ FW, respectively. Asl-e-Qarabagh was observed to have the least H_2O_2 content (0.042 $\mu\text{g/g}$ FW) among cultivars. Again, in inoculated female flowers, those with the highest concentration of proline (Mish-Pestan and Tabestaneh) were found to have the highest H_2O_2 content (0.335 and 0.331 $\mu\text{g/g}$ FW, respectively, Figure 5). Hydrogen peroxide increases are due to initiate signaling systems as a messenger to activate the cold resistance genes, however, excessive increase of this enzyme triggers an enhancement in catalase and peroxidase to quench it. When plants are exposed to cold stress, due to disturbance in plant metabolism, the production of oxygen radicals such as superoxide ($-O_2$), hydrogen peroxide, and hydroxyl ($-OH$) increase (Fasih and Afshari, 2018; Rasoulnia *et al.*, 2011).

Klíma *et al.* (2012) investigated the reaction of different parts of *Larix europea* under different temperature treatments. The result indicated a different pattern of peroxidation activity in leaf, seed, and branch samples, and peroxidation activity of leaf samples were several folds higher than branch samples. Research on *Quercus robur* has shown that with seasonal changes, peroxidase activity fluctuates so that its activity increases at the beginning of the cold season (Morecroft *et al.*, 2003) and with the approach of the cold season, peroxidase activity has increased and in December, several times higher than in summer. However, its amount has decreased slightly compared to September. Castillo (1986) and Hung and Kao (2004) showed that peroxidase is the most sensitive plant enzyme to environmental stresses. Examination of black pine branches showed that peroxidase activity was not the same in different seasons and was several times higher in the winter (Chen *et al.*, 2006).

Peroxidase activity was not the same in different seasons of the year, its maximum activity is in the cold seasons of the year while it was the least in the summer (Siqueira *et al.*, 2007). Investigating the relationship between peroxidase activity and phloem vascularization in pine, spruce, and birch stem during the growing season showed that peroxidase activity increases at the beginning of the growing and lignification period (Marjamaa *et al.*, 2003). The earlier the small amount of this enzyme increases at the beginning of the cold season, the greater its resistance to

early frosts, and also the less it decreases in early spring compared to winter, indicating its higher resistance to the occurrence of early spring frosts (Fischer and Höll, 1991; Zolfaghari *et al.*, 2005). At low temperatures, hydrogen peroxide accumulates without decomposing due to the inactivity of the enzyme catalase. During the freezing stages, respiration often increases and then begins to decrease (Prasad *et al.*, 1994; Purvis and Shewfelt, 1993).

4. CONCLUSIONS:

In the seasonal changes of proline observed in this study, the accumulation of this amino acid during the cold adaptation period increased the resistance. The aggregation of this amino acid in Mish-Pestan and Tabestaneh cultivars were found to be the highest. Generally, it can be inferred that Mish-Pestan and Tabestaneh owe their cold resistance to a large extent to the accumulation of proline. The fluctuation of hydrogen peroxide concentration under cold treatments observed in this study was not significant compared to proline. Therefore, with very small changes in this enzyme, particularly in Mish-Pestan and Tabestaneh cultivars, an increase in cold resistance in those two cultivars was observed. In areas where there is a risk of freezing, hazelnut-resistant cultivars should be cultivated, which among the eight cultivars studied, Mish-Pestan and Tabestaneh cultivars were more resistant. Given the importance of this crop and its vulnerability to freezing, it is highly recommended that other cultivars be comprehensively investigated to find the cold stress tolerance cultivars and realize the underlying physiological mechanisms involved.

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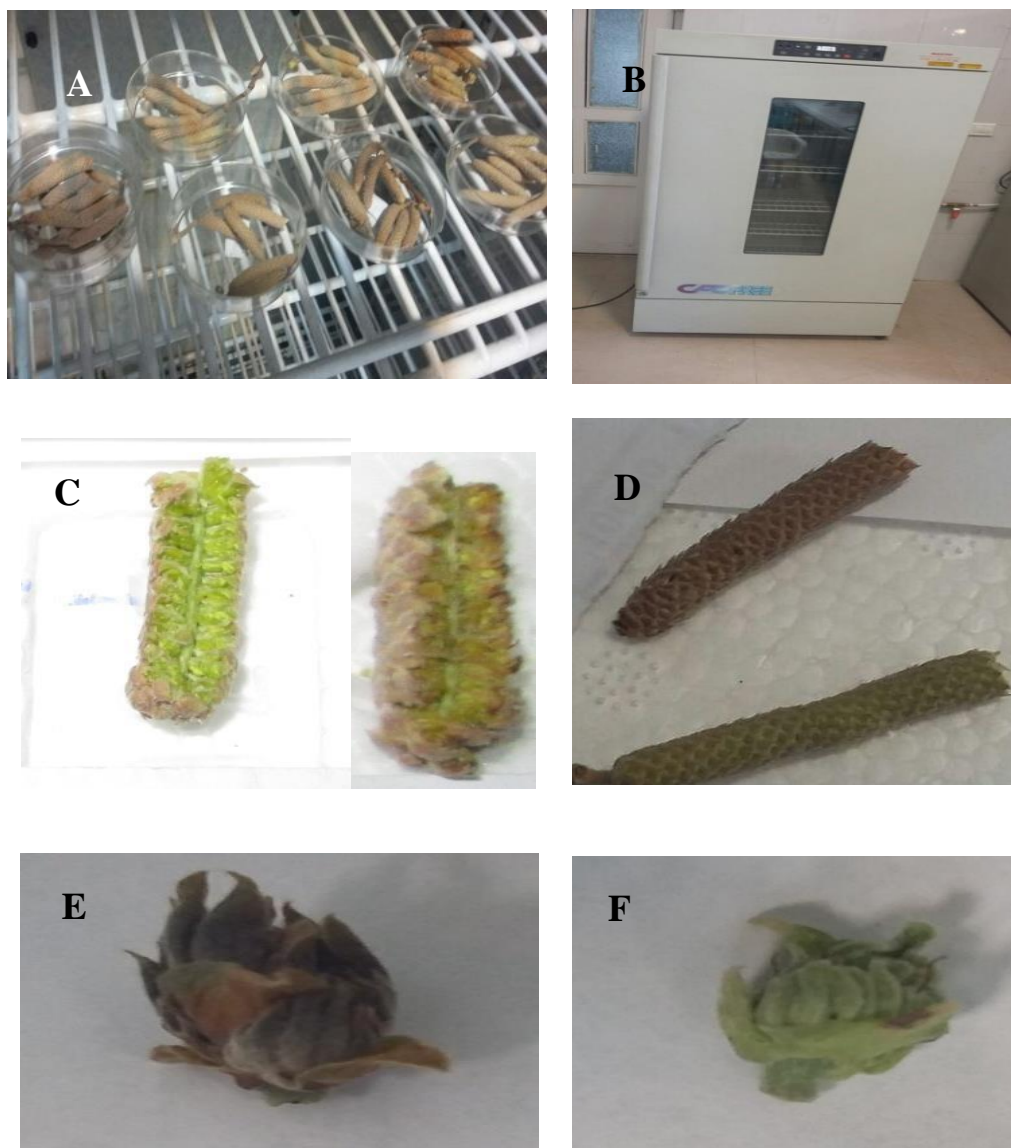


Figure 1. (A and B) Samples of unopened male catkins and inoculated female flowers of eight hazelnut cultivars prepared and placed in the incubator to be exposed to low temperature. (C and D) male catkins under normal temperature and injured by cold stress, respectively. (E and F) inoculated female flowers as control and damaged by cold stress, respectively.

Table 1. Scoring qualitative morphological traits of male catkins exposed to low-temperature stress.

	zero (no change)	one (low change)	three (medium change)	five (high change)	seven (very high change)
Cultivars	No changes	The decreased freshness of tissues, no external or internal discoloration	Reduction in tissue hardness, the onset of freezing, no external or internal discoloration	Reduction in tissue hardness, freezing of tissues, external discoloration from green to dark green	Reduction in tissue hardness, complete freezing, and external and internal discoloration to brown
Nakhnroud	+1,+3	-1	-3	-5	-7
Khandan	+1,+3	-1	-3	-5	-7
Tabestaneh	-1,+1,+3	-3	-5	-7	-7
South of Qarabagh	+1,+3	-1	-3	-5	-5
Asl-e-Qarabagh	+1,+3	-1	-3	-5	-7
Rasmi	+1,+3	-1	-3	-5	-5
Gerdashkevar	+1,+3	-1	-3	-5	-5
Mish-Pestan	+1,+3	*	-1,-3	-5	-5

Table 2. Scoring qualitative morphological traits of inoculated female flowers exposed to low-temperature stress.

	Zero (no change)	one (low change)	three (medium change)	five (high change)	seven (very high change)
Cultivars	No changes	Weak external discoloration	Weak external discoloration, the onset of freezing	Weak external browning, freezing, weak internal discoloration	Freezing, external and internal discoloration (Dark green), and necrosis
Nakhnroud	-1,+1,+3	-3	-5	-7,-9	-11
Khandan	+1,+3	-1	-3	-7,-5	-11,-9
Tabestaneh	+1,+3	-1	-5,-3	-9,-7	-11
South of Qarabagh	+1,+3	-3,-1	-5	-9,-7	-11
Asl-e-Qarabagh	+1,+3	-1	-5,-3	-7	-9
Rasmi	+1,+3	-1	-5,-3	-9,-7	-11
Gerd-e-h-ashkevar	+1,+3	-1	-3	-7,-5	-9
Mish-Pestan	-3,-1,+1,+3	-5	-7	-9	-11

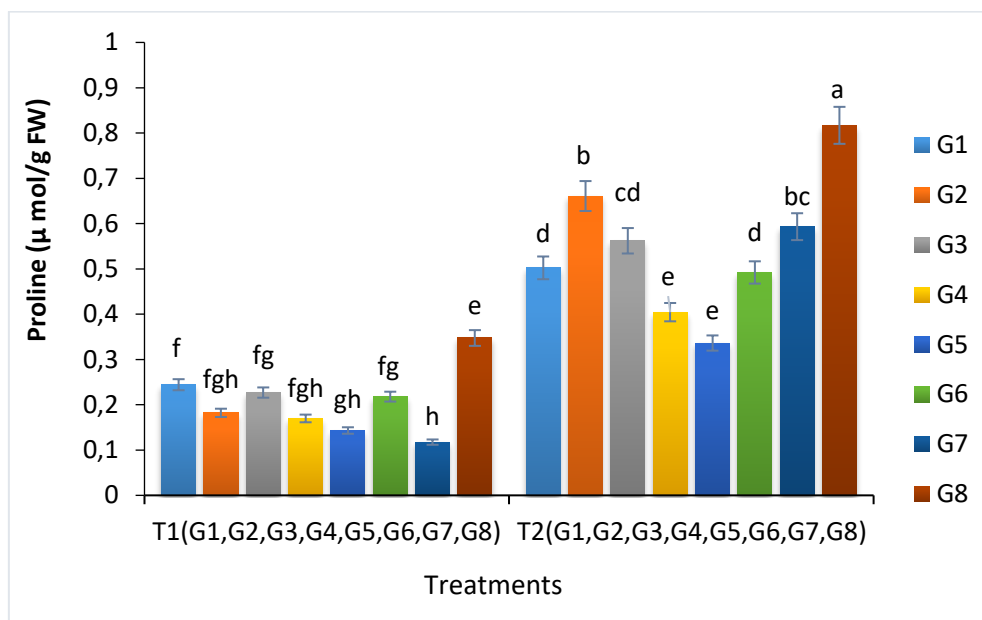


Figure 2. The effect of cold stress treatments on proline accumulation in male catkins of hazelnut cultivars. **Note:** Cultivars: G1; Nakhnroud, G2; Khandan, G3; Tabestaneh, G4; South of Qarabagh, G5; Asl-e-Qarabagh, G6; Rasmi, G7; Gerdashkevar, and G8; Mish-Pestan

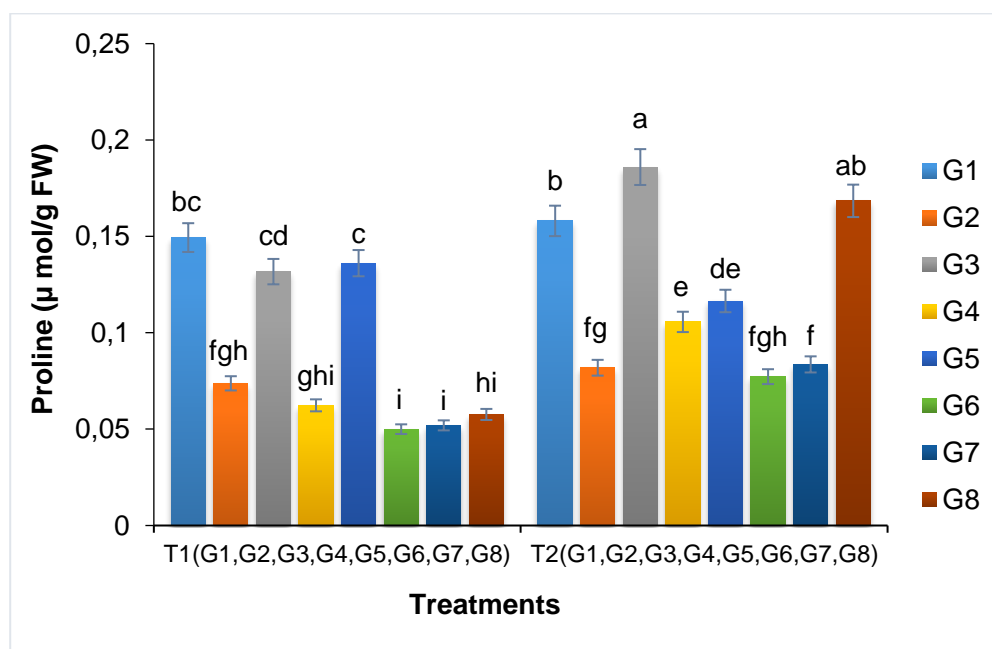


Figure 3. The effect of cold stress treatments on proline accumulation in inoculated female flowers of hazelnut cultivars. **Note:** Cultivars: G1; Nakhnroud, G2; Khandan, G3; Tabestaneh, G4; South of Qarabagh, G5; Asl-e-Qarabagh, G6; Rasmi, G7; Gerdashkevar, and G8; Mish-Pestan

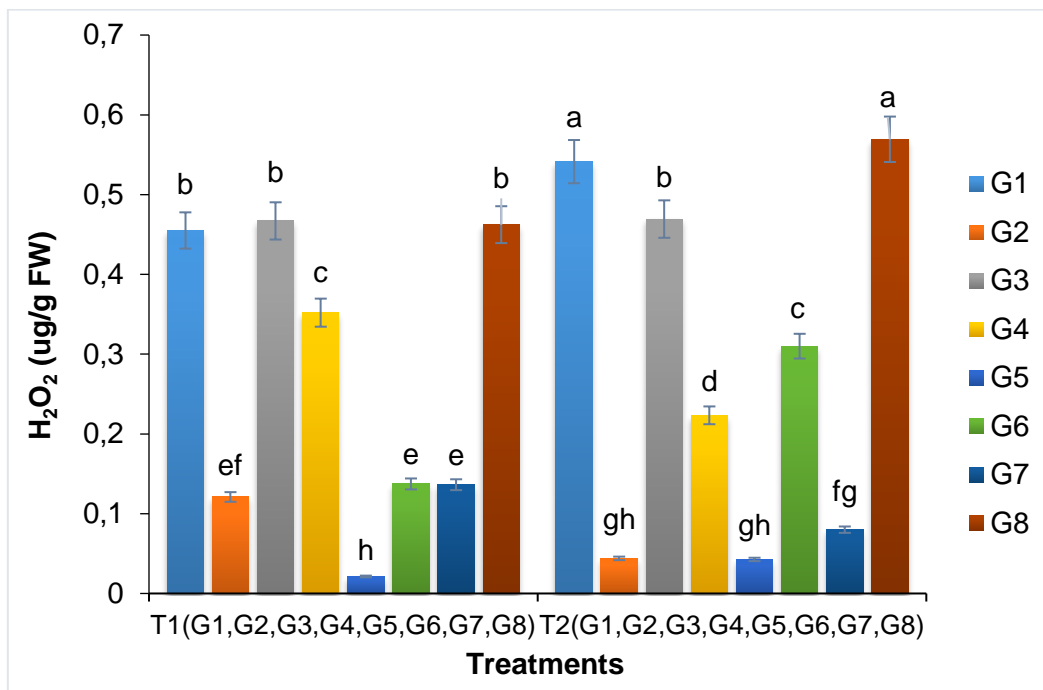


Figure 4. The effect of cold stress treatments on H_2O_2 accumulation in male catkins of hazelnut cultivars. **Note:** Cultivars: G1; Nakhnroud, G2; Khandan, G3; Tabestaneh, G4; South of Qarabagh, G5; Asl-e-Qarabagh, G6; Rasmi, G7; Gerdashkevar, and G8; Mish-Pestan.

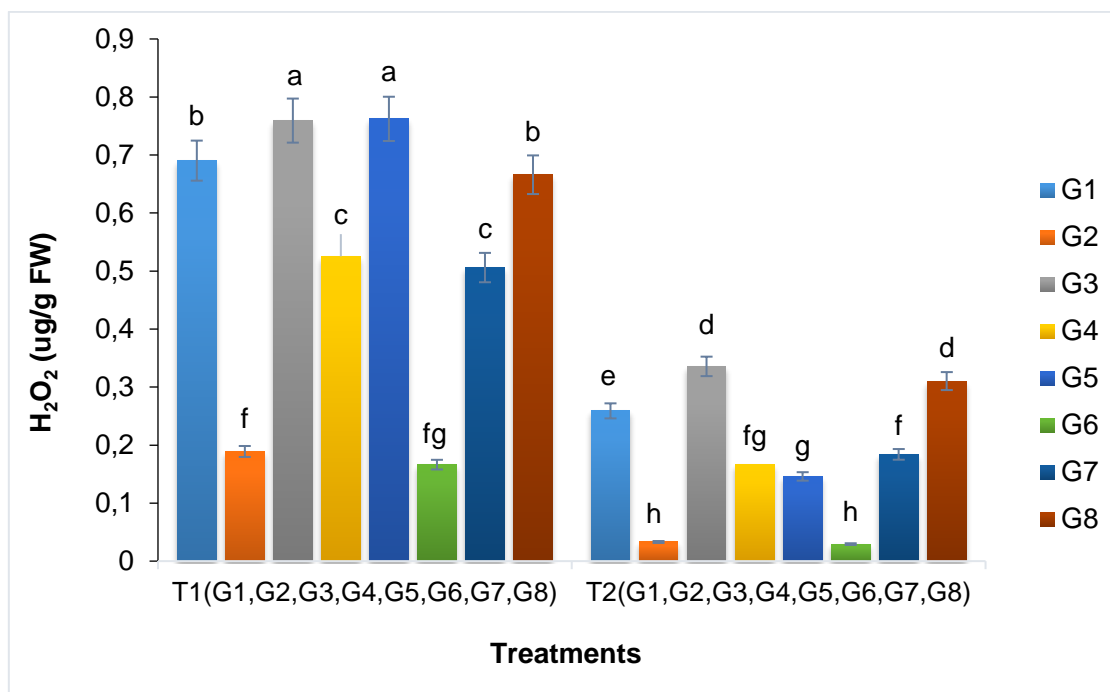


Figure 5. The effect of cold stress treatments on H_2O_2 accumulation in inoculated female flowers of hazelnut cultivars. **Note:** Cultivars: G1; Nakhnroud, G2; Khandan, G3; Tabestaneh, G4; South of Qarabagh, G5; Asl-e-Qarabagh, G6; Rasmi, G7; Gerdashkevar, and G8; Mish-Pestan

CALCULATION FOR REDEMPTION OF COMPACT TESTING BY THE PROCTOR METHOD THROUGH NEWTON'S GRAVITATIONAL POTENTIAL ENERGY.

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ABSTRACT

Throughout this article, a study on the characteristics of the compaction test by the Proctor Method, regulated by ABNT NBR-7182, which is used to verify the degree of soil compaction, will be approached in order to broaden the discussion and raise points that demonstrate the urgent need to make it more accurate, efficient and safe. Through qualitative and quantitative research carried out by the authors of this article, it sought to collect data through a questionnaire for professionals in the field of geotechnics in the “Quadrilátero Ferrífero” region in Minas Gerais. In addition to other relevant data for the topic, it was raised that of the 22 professionals from the participating region, 72.7% of the total belief that the manual compaction test can be manipulated by an operator during the test execution, failing to generate results reliable, thus showing the importance of the proposed theme. In this way, we initially sought to correlate the Compaction Energy formula idealized by Ralph Proctor with Isaac Newton's Gravitational Potential Energy formula and, through it, present the resizing, which may enable the construction of manual, semi-automatic human propulsion machines (not or making the automated ones that depend on electricity available to the market. In conclusion, from the mathematical calculations, it was possible to evidence the use of Newton's Gravitational Potential Energy to construct new equipment to carry out this test.

Keywords: *civil construction, compaction test, proctor test.*

1. INTRODUÇÃO:

É fato que em qualquer obra da construção civil onde o terreno passou por processos antrópicos, ou seja, com interferência humana, é necessária a compactação desse solo, o que foi discutido também no trabalho de Pivetta (2017). A compactação é definida como sendo o adensamento dos solos pela aplicação de energia mecânica (Holtz and Kovacs, 1981). Em seu trabalho, Hillel (1998) afirma que a compactação do solo pode ser considerada do ponto de vista da engenharia civil ou da agronomia. Para engenharia civil, o método utilizado atualmente para verificar se o solo foi compactado corretamente é o método de Proctor, previsto na ABNT NBR-7182 que tem como objetivo “prescrever o método para determinar a relação entre o teor de umidade e a massa específica aparente seca de solos quando compactados, de acordo com os processos especificados”.

O método de Proctor foi criado em 1933 pelo engenheiro Ralph Proctor, considerado por muitos como o Patrono da Geotecnia Mundial. Ele propôs a criação da energia de compactação que é o “ato de artificialmente aumentar o peso

específico do solo por manipulação agindo-se sob forma de pressão ou vibração das partículas de solo de modo que elas fiquem em estado de contato íntimo” (Baptista, 1974). Porém, nota-se o quão antigo é o método, uma vez que desde o seu desenvolvimento este ensaio utiliza os mesmos princípios apresentados. Portanto, é fundamental a democratização e aprimoramentos desse ensaio, visando torná-lo mais preciso, ágil e seguro.

O processo de compactação do solo tem como objetivo reduzir o volume, ou seja, retirando o ar contido em um solo, tornando-o mais estável e resistente, além disso, visa a melhoria na permeabilidade, absorção de água e compressibilidade (Caputo, 2015; Murrieta, 2018). A compactação resulta em um aumento na resistência ao cisalhamento e resistência à carga, redução na deformabilidade e permeabilidade (Crispim, 2010), além de reduzir a sedimentação do solo e dar estabilidade.

Contudo, a realização do ensaio citado depende diretamente da interferência humana, como discutido também por Silva e Andrade (2016) uma vez que os testes automatizados

existentes são lentos, causando uma tendência do uso do processo manual, porém essa dependência gera oportunidade para que ocorram erros ou manipulações em parâmetros durante o ensaio, causadas de forma intencional ou não pelo laboratorista, em relação à altura, operação do soquete, número de camadas, número de golpes por camadas, gerando divergências com as determinações que a norma prevê, e desse modo, haverá uma imprecisão inadmissível, podendo até mesmo ocasionar acidentes graves no futuro.

Portanto, pretendendo tornar a compactação do solo cada vez mais segura, o presente trabalho tem como intuito levantar a discussão sobre o método de compactação de Proctor, seja com suas variações de energia normal, intermediário ou modificado, apontando as possíveis ocorrências de erros por parte do laboratorista no momento da realização do ensaio, seja intencional ou pelo cansaço físico. Além disso, será proposto também o redimensionamento dos soquetes utilizados na execução do ensaio de compactação pelo método de Proctor, tendo como base a energia potencial gravitacional criada por Newton, possibilitando assim a criação de máquinas mais tecnológicas, que façam os ensaios se tornarem mais precisos, ágeis e seguros.

É importante frisar que, segundo matéria divulgada pela Revista Galileu em março de 2020, Isaac Newton descobriu a Lei da Gravitação Universal durante a epidemia da peste bubônica, que afetou a Inglaterra entre 1665 e 1666. Na época a Universidade de Cambridge, onde estudava, liberou seus alunos para entrarem em quarentena, portanto foi nesse período que Newton colocou seus estudos em andamento, alguns de seus biógrafos afirmam que foi nessa época que ele teve o possível *annus mirabilis*, latim para "ano miraculoso" ou "ano de maravilhas". (Lança, 2005).

Por coincidência, este trabalho também foi escrito durante uma quarentena de pandemia. Os autores utilizaram da internet para se comunicarem e organizar conjuntamente o artigo, respeitando o isolamento social, trabalhando a distância e se protegendo do Coronavírus, ressaltando assim que o estudo e confecção do artigo só foi possível graças aos antigos estudos de Newton durante uma quarentena e também por causa das atuais tecnologias de comunicação que tornaram possível a comunicação entre os autores.

Vale evidenciar ainda que, existem

máquinas que realizam os ensaios de compactação (Lobo, 2003), porém é necessário um grande investimento para as empresas adquirirem essas máquinas de compactação, principalmente por causa do valor elevado. Fato que contribui negativamente e faz com que as máquinas não estejam em atividade nas frentes de serviço de terraplanagem no Brasil.

O que o atual trabalho propõe é que seja realizada uma urgente padronização da energia de compactação no método de Proctor e demais ensaios derivados, seguindo a tendência do que foi discutido no trabalho de Tartarotti *et al.* (2018), que com a tendência da Indústria 4.0 cada vez mais as decisões serem tomadas sem interferência humana, de maneira a garantir uma maior confiabilidade. É fundamental ressaltar a originalidade do presente trabalho ao propor o redimensionamento dos instrumentos do ensaio para que seja desenvolvido máquinas mais baratas, portáteis e acessíveis ao mercado. Além disso, a maneira com que os cálculos são apresentados, não é encontrado em outra fonte acadêmica.

2. MATERIAIS E MÉTODOS:

2.1. Ensaio de Compactação de Proctor

O ensaio de compactação, criado por Ralph Proctor em 1933 foi desenvolvido para uso na engenharia civil, cujo propósito é alcançar a máxima compactação de um terreno onde irá ser construída uma edificação (Vargas, 1977). Portanto, ele foi criado com intuito de determinar o momento em que o solo está mais suscetível à compactação, ou seja, com a umidade dentro dos parâmetros definidos. Com o ensaio de Proctor, é possível chegar na curva de compactação por meio da análise dos valores de umidade do solo com a densidade. Entretanto, para isso é necessário que a energia de compactação seja a mesma em todas as quedas. (Braida *et. al.*, 2006)

Proctor determinou três energias de compactação, elas são divididas em: normal, intermediária e modificada (Santos, Daibert; 2018). Conforme foi apresentado na Tabela 1, cada energia possui um número de golpes, número de camadas e tipo de soquete específico. A execução do ensaio deve ser realizada conforme a norma prescreve, sendo que energia de compactação normal equivale a 59 N.cm/cm³, a intermediária 130 N.cm/cm³ e a modificada 270 N.cm/cm³. (ABNT NBR 7182)

O ensaio de compactação é realizado com

os seguintes equipamentos: cilindro metálico pequeno, cilindro metálico grande, soquete pequeno, soquete grande, balanças, peneiras, estufa, cápsulas metálicas, bandejas metálicas, régua de aço biselada, espátulas de lâmina flexível, provetas de vidro, desempenadeira de madeira, extrator de corpo-de-prova, conchas metálicas, base rígida de concreto, papel filtro, conforme a norma ABNT NBR 7182.

Com os equipamentos supracitados, o laboratorista tem a possibilidade de realizar os ensaios de compactação pelo método de Proctor de forma manual. No Proctor Normal, o operador deve retirar do solo a quantidade de terra necessária, em seguida, esses resíduos passam por uma peneira de 4,8 mm. Adiciona-se água na amostra, até notar a consistência correta, compacta-se utilizando o cilindro pequeno com 3 camadas, aplicando 26 golpes por camadas a uma altura de 0,305 m. Retira-se o colarinho e a base, utilizando a régua biselada aplaina-se a superfície na altura do molde, após isso, faz-se a pesagem do conjunto. Com o auxílio do extrator retira-se a amostra do molde, e coleta-se uma pequena quantidade para determinação da umidade, o restante do material é desmanchado até que possa passar novamente pela peneira de 4,8 mm e guardado junto ao restante da amostra inicial. Adiciona-se água à amostra homogeneizando-a, normalmente a uma ordem de 2% da massa original do solo, repete-se o processo por quatro vezes. Por último, faz-se os cálculos necessários para encontrar a curva de compactação, peso específico, umidade ótima e as curvas de saturação (ABNT NBR 7182).

2.1.1 Problemas relacionados ao ensaio

Os autores do atual trabalho realizaram entre os meses de março a abril de 2020 entrevistas com auxílio do Google Forms, cujo intuito principal foi constatar através da opinião dos profissionais que operam os ensaios na região, a possibilidade de manipulação dos resultados durante a execução. Ao todo, participaram dessa pesquisa, 22 profissionais que atuam na área da construção civil e geotecnia que serão apresentados nos resultados deste trabalho.

Além disso, conforme o trabalho de Crispim *et. al* (2015), neste tipo de ensaio faz-se necessário avaliar a influência do diâmetro e do número de camadas do corpo de prova nas curvas de compactação de laboratório e na resistência mecânica dos solos.

O trabalho de Euqeres, Arão e Lima

(2018) levanta a problemática referente a preocupação com a ergonomia na realização das atividades desenvolvidas no ambiente de Laboratório de Solos, pois grande parte dessas dependem não somente de esforços físicos, mas a combinação deste com número significativo de repetições. Conforme levantado pelas autoras, a compactação de corpos de prova poderia ser realizada de forma mecanizada, porém como os equipamentos para tal atividade podem ser algo que torne a atividade bastante onerosa para o contratante, a compactação de forma manual ainda é muito utilizada.

2.1.2 Correlação entre a Fórmula da Energia de Compactação com a Fórmula de Potencial Gravitacional

O presente trabalho propõe o redimensionamento dos instrumentos, principalmente do soquete que realiza os ensaios, que possibilitará a construção de máquinas acessíveis ao mercado, colocando fim a imprecisão dos resultados obtidos pelo ensaio de compactação pelo método de Proctor realizado de forma manual.

A energia potencial gravitacional consiste em fazer a multiplicação da massa, gravidade e altura da partícula analisada (Equação 1). (Marques, 2016).

$$E_{pg} = m \times g \times h \quad (\text{Eq. 1})$$

Onde: E_{pg} (Energia potencial gravitacional); m (Massa); g (Gravidade);

Ralph Proctor postulou que energia de compactação é obtida por meio da multiplicação do peso, altura, número de camadas e o número de golpes, e dividindo o resultado dessa multiplicação pelo volume (Equação 2).

$$E_c = (P \times h \times N \times n) / v \quad (\text{Eq. 2})$$

Onde: E_c (Energia de compactação); P (Peso); h (Altura); N (Número de camadas); n (Número de golpes); v (Volume).

Desmembrando o peso na fórmula da energia de compactação, obtém-se a fórmula da

energia de compactação modificada que consiste na multiplicação da massa, gravidade, altura de queda do tarugo, número de golpes exercido, e número de camadas no cilindro, e o resultado dessa multiplicação é dividido pelo volume (Equação 3).

$$E_{cm} = (m \times g \times h \times N \times n) / v \quad (\text{Eq. 3})$$

Onde: E_{cm} (Energia de compactação modificada); m (Massa); g (Gravidade); h (Altura); N (Número de camadas); n (Número de golpes); v (Volume).

Contudo, ao observar essa fórmula é possível verificar que o início dela é igual à fórmula de energia potencial gravitacional. Ou seja, por meio dessa correlação que é possível realizar o redimensionamento.

2.1.3 O Redimensionamento

Após realizar essa análise de correlação, é notável a possibilidade do redimensionamento dos instrumentos do ensaio de compactação pelo método de Proctor. Na energia de compactação Normal, é determinado que o volume do cilindro seja em média de 1000 cm³, a massa do tarugo seja de 2,5 Kg, e que a altura em queda livre seja de 30,5 cm (conforme a Tabela 2 apresenta). Desse modo, ao realizar os cálculos com a fórmula de energia de compactação temos sempre que obrigatoriamente chegar a um valor de 59,475 N.cm/cm³, valor esse determinado por Proctor como energia Normal de compactação. Vale ressaltar que Proctor utilizou a gravidade valendo 10 m/s² ao invés de 9,80 m/s², portanto, visando maior segurança para o redimensionamento, respeitando a padronização proposta por Proctor, neste trabalho também será considerado a gravidade como 10 m/s².

Desse modo, é necessário calcular a energia potencial gravitacional, com a massa e altura determinada por Proctor. Para isso, pega-se a fórmula da energia potencial gravitacional (Equação 4), substitui os dados de massa e altura (Equação 5) e faz a multiplicação (Equação 6). Ao realizar esse cálculo obterá que a energia potencial gravitacional no Proctor Normal equivale a 7,625 J.

$$E_{pg} = m \times g \times h \quad (\text{Eq. 4})$$

$$E_{pg} = 2,5 \times 10 \times 0,305 \quad (\text{Eq. 5})$$

$$E_{pg} = 7,625 \text{ J} \quad (\text{Eq. 6})$$

Portanto, para realizar o redimensionamento do ensaio de compactação por Proctor Normal, basta utilizar o valor que foi encontrado acima para substituir a energia potencial gravitacional juntamente com os dados determinados por Proctor, e aplicá-los utilizando a fórmula da energia potencial gravitacional (Equação 7).

Sendo que, poderá modificar a massa ou a altura, caso utilize um novo valor de massa, proporcionalmente encontrará uma nova altura ou se colocar uma nova altura encontrará proporcionalmente um novo valor para massa.

No exemplo abaixo, supõe que a nova massa fosse de 5,5 Kg, ou seja, esse valor foi inserido na fórmula da energia potencial gravitacional juntamente com 7,625 J (Equação 08), que foi o valor encontrado no Proctor Normal. Após realizar as operações matemáticas, obtém-se que nova altura terá que ser de 13,86 cm para que a energia potencial gravitacional seja a mesma que Ralph Proctor padronizou no Proctor Normal (Equação 10).

$$E_{pg} = m \times g \times h \quad (\text{Eq. 1})$$

$$7,625 = 5,5 \times 10 \times h \quad (\text{Eq. 8})$$

$$7,625 = 55h \quad (\text{Eq. 9})$$

$$h = 13,86 \text{ cm} \quad (\text{Eq. 10})$$

Desse modo, foi possível redimensionar o ensaio de compactação, determinando novos valores de massa e altura, no exemplo acima foi determinado que para uma massa de 5,5 kg é necessário cair em queda livre de uma altura de 13,86 cm, a fim de que a energia de compactação seja a mesma postulada por Proctor.

Portanto, para uma validação matemática, basta colocar os dados encontrados na fórmula da energia de compactação modificada (Equação 11), e chegará no mesmo valor determinado por Proctor (Equação 12).

$$E_{cm} = (7,5 \times 10 \times 10,17 \times 3 \times 26) \div 1000 \quad (\text{Eq. 11})$$

$$E_{cm} = 59 \text{ N.cm/cm}^3 \quad (\text{Eq. 12})$$

O valor da energia potencial gravitacional

para o ensaio de compactação pelo método de Proctor com cilindro de volume 2000 cm³, será o mesmo para todos os tipos de ensaios com energia Normal, Intermediária ou Modificada. Ou seja, utiliza-se a fórmula da energia potencial gravitacional (Eq. 13), substitui-se os valores de massa e altura conforme Proctor determinou (Eq. 14), e resultará no valor da energia potencial gravitacional para os três tipos de energias. Portanto, a diferença entre os tipos de energias nesse caso, é apenas a variação de número de golpes em cada ensaio. Entretanto, é necessário entender que por não ter alteração na altura de queda livre e na massa do tarugo, a energia potencial gravitacional em cada golpe é igual nesses três tipos de energias de compactação.

$$E_{pg} = m \times g \times h \quad (\text{Eq. 1})$$

$$E_{pg} = 4,5 \times 10 \times 0,45 \quad (\text{Eq. 14})$$

$$E_{pg} = 20,25 \text{ J} \quad (\text{Eq. 18})$$

Portanto, com o valor de 20,25 J encontrado é possível redimensionar o ensaio de Proctor Normal de volume 2000 cm³, Intermediário e o Proctor Modificado. Para isso basta determinar ou a massa ou a altura e substituí-los na fórmula da energia potencial gravitacional. Na Tabela 4, é possível encontrar os valores da energia potencial gravitacional para o redimensionamento do ensaio de compactação de Proctor.

3. RESULTADOS E DISCUSSÕES:

Conforme apresentado na Tabela 3, nota-se que uma grande maioria dos participantes da pesquisa realizada pelos autores, afirmam que os ensaios de compactação realizados de forma manual podem apresentar erros.

Ademais, é evidente observar ainda na Tabela 3 que a disponibilidade de máquinas que realizam os ensaios de compactação nas empresas é quase nula, visto que 100% dos entrevistados disseram que onde trabalham não há nenhuma máquina que tenha esse objetivo. Sabe-se que atualmente existem máquinas que atendem essa área, porém pelo fato delas terem peso e tamanhos elevados dificulta a locomoção e são custosas para as empresas. Além disso, o suporte técnico é insuficiente em vários locais do mundo, fator que geraria ainda mais despesas para as empresas.

Nesta mesma pesquisa foi deixado um

campo para que os participantes respondessem de que maneira poderia haver ocorrências de erros humanos durante o ensaio de compactação, resultado está exposto no Gráfico 1. Vale ressaltar que a pergunta foi utilizada para gerar dados qualitativos, ou seja, as respostas foram pessoais e abertas. Portanto, os valores em porcentagem apresentados, se somados, não alcançará a totalidade de 100%, pois houve respostas que continha mais de uma ocorrência.

Desse modo, analisando os dados do Gráfico 1, nota-se que, cerca 36,40% das respostas dos participantes acreditam que a maior causa de manipulação no ensaio de compactação se deve ao erro de exercer força no soquete no momento da queda. E tendo um empate na segunda e terceira colocação, com 13,60%, o número de camadas diferentes do estabelecido e alteração nos resultados finais do ensaio.

Diante dos dados supracitados, é visível que o ensaio de compactação de forma manual abre brechas para que ocorram manipulações na sua realização e, portanto, essa situação pode ocasionar grandes acidentes no futuro. Além disso, como citado no trabalho de Sobreira *et. al.* (2018), o ensaio Proctor, não evoluiu muito desde a sua concepção. Desta forma, pesquisas sobre métodos de compactação de solos em laboratório vêm sendo retomadas com o intuito de buscar um mecanismo de ação que simule de forma mais semelhante a compactação de campo, se comparado ao ensaio Proctor.

É notável que os problemas apresentados no atual trabalho em relação aos ensaios de compactação realizados de forma manual são prejudiciais à segurança, pois possuem baixa confiabilidade. Nesse contexto, as pesquisas realizadas por meio da ferramenta do Google Forms com trabalhadores da área da construção civil e geotecnia, corroboram para tal análise, visto que conforme a Tabela 3, cerca de 72,7% dos entrevistados afirmaram que acreditam que o ensaio de compactação de forma manual pode ser manipulado. Contudo, todas as lacunas apresentadas levaram o presente artigo a propor uma solução, visando tornar os ensaios mais precisos, ágeis, seguros e ergonomicamente melhor para os operadores.

Por meio da correlação entre as fórmulas da energia de compactação e da energia potencial gravitacional, foi detectado a possibilidade do redimensionamento. Portanto, conforme os dados apresentados na Tabela 4 isso é realizável, ou seja, através dos valores 7,25 J e 20,25 J encontrados por meio de cálculos, é possível o

redimensionamento nos quatro tipos de energia de compactação, sendo que o valor de 7,25 J é utilizado para redimensionar o Proctor Normal, e o valor de 20,25 J redimensiona o Proctor Normal de volume 2000 cm³, Intermediário e o Proctor Modificado. Contudo, basta substituir na fórmula da energia potencial gravitacional os valores citados acima e determinar qual o novo valor da massa ou da altura, dado esse que são inversamente proporcionais.

Desse modo, o presente redimensionamento abre possibilidade para a construção de máquinas semiautomatizadas, ou seja, que realiza os ensaios de compactação com baixa interferência humana. Fato esse que fornece resultados mais seguros, aumentando a confiabilidade dos ensaios.

4. CONCLUSÕES:

A partir da correlação apresentada no atual trabalho que possibilita recriar os mecanismos que são utilizados para este ensaio tão importante na construção civil, trazendo novas possibilidades de equipamentos e redução da interferência humana no ensaio. Com os resultados do estudo apresentado é possível afirmar que a realização do ensaio de compactação pelo método de Proctor de forma manual pode apresentar possíveis imprecisões, pois os dados coletados de forma equivocada ou até mesmo de forma intencional durante o ensaio, poderá ocasionar grandes problemas nas estruturas das obras e possíveis acidentes no futuro.

Portanto, é notável que o redimensionamento do ensaio de compactação pelo método de Proctor como foi proposto pode contribuir para a construção de máquinas, seja manuais ou automáticas, cujo objetivo seja realizar o ensaio de compactação com a menor interferência humana possível, como foi exposto, tornando-o mais seguro, ágil e preciso.

Além disso, verificou-se que a partir dos cálculos matemáticos é possível modificar o tamanho das peças sem alterar o valor da energia de compactação. Vale ressaltar ainda que os valores apresentados na Tabela 4 que norteará a realização dos redimensionamentos propostos. Desse modo, com o redimensionamento apresentado é viável à criação de máquinas acessíveis ao mercado, ou seja, que são menores e de baixo custo, diferentemente dos equipamentos existentes que realizam os ensaios de compactação, que demanda das empresas

grandes investimentos.

Ademais, entende-se que a área da geotecnia é muito importante para manter a segurança das edificações. Portanto, é fundamental que novos estudos sobre o assunto continuem sendo publicados, para que haja ampla discussão sobre a geotecnia.

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Tabela 1 - Energia de Compactação ABNT (NBR 7182)

Cilindro	Características inerentes a cada energia de compactação.	Energia		
		Normal	Intermediária	Modificada
Pequeno	Soquete	Pequeno	Grande	Grande
	Número de camadas	3	3	5
	Número de golpes por camada	26	21	27
Grande	Soquete	Grande	Grande	Grande
	Número de camadas	5	5	5
	Número de golpes por camadas	12	26	55
	Altura do disco espaçador (mm)	63,5	63,5	63,5

Tabela 2 - Energia de compactação por impacto.

Designação	Peso (N)	Altura de queda (cm)	Número de camadas	Número de Golpes	Volume do Cilindro (cm ³)	Energia (N.cm/cm ³)
Proctor Normal (PN)	25	30,5	3	26	1.000	59
Proctor Normal (PN)	45	45,0	5	12	2.000	60
Intermediário (PI)	45	45,0	5	26	2.000	130
Proctor Modificado (PM)	45	45,0	5	55	2.000	270

Tabela 3. Questionário aplicado a profissionais que realizam ensaio de compactação.

Perguntas	Sim	Não
Na empresa onde trabalha é realizado o ensaio de compactação?	90,9%	9,1%
Na empresa onde trabalha tem compactador de solo automático?	0%	100%
Uma máquina de compactação de solo automática facilitaria seu serviço?	85,7%	14,3%
Você acredita que o ensaio de compactação de solo pode ser manipulado por um operador?	72,7%	27,3%

Tabela 4 - Energia Potencial Gravitacional para o Redimensionamento do Ensaio de Compactação

Designação	Energia Potencial Gravitacional para o Redimensionamento (J)	Volume (cm ³)	Energia de Compactação (Ncm/cm ³)
Proctor Normal (PN)	7,625	1000	59
Proctor Normal (PN)	20,25	2000	60
Proctor Intermediário (PI)	20,25	2000	130
Proctor Modificado (PM)	20,25	2000	270

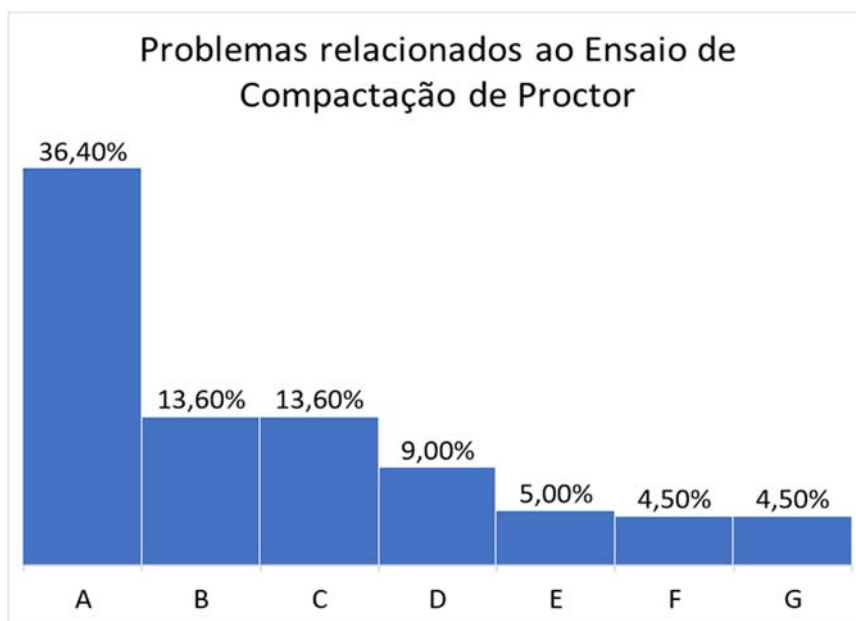


Gráfico 1 – Problemas relacionados ao Ensaio de Compactação de Proctor

LEGENDA	
A	Erro ao exercer força no soquete
B	Número de golpe por camada diferente do estabelecido pela norma;
C	Alteração nos resultados no final do ensaio;
D	Erro na quantidade de água adicionada ao solo;
E	Não responderam.
F	Erro no número de camadas;
G	Não sabem;

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ABSTRACT

The extraction of naturally occurring compounds is one of the fastest-growing industries because of its benefits against its synthetic analogs. Environmental protection must require the use of natural products instead of chemicals to minimize pollution. Thus, this investigation studies the use of some natural product, as curcumin, as naturally occurring acid-base indicators. Curcumin can be used as acid-base indicators since it was found that it possesses pH-dependent solubility. Curcumin, the major active component of turmeric, *Curcuma longa* (Zingiberaceae), is used as a spice in curry and as a coloring agent in yellow mustards, cosmetics, pharmaceuticals, and hair dyes. In this research, the main compound colored rhizome of turmeric (*Curcuma longa*) cultivated in Mérida, Venezuela, is extracted: Curcumin (C₂₁H₂₀O₆) (1*E*,6*E*)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione, in a yield of 3.42% after 8 hours of extraction using soxhlet extractor system with organic solvents (hexane and ethanol). The thin-layer chromatography and column performed separation and purification using a mobile phase, a mixture of chloroform-hexane 3:2. The dye was characterized by spectroscopic analysis of visible ultraviolet (UV-Vis) and infrared (IR), in addition to his studio in steering sensitivity as an acid-base indicator. This dye is useful as an acid-base indicator in strong acid-strong base volumes and did not require large amounts of it as it has high sensitivity. The results indicate that curcumin as an acid-base indicator allows the development of new standards in different chemistry fields that require this type of analysis.

Keywords: *Curcuma longa*, curcumin, acid-base indicator, soxhlet extraction, chromatography.

1. INTRODUCTION:

Of the existing acid-base indicators used in laboratories, the so-called synthetic indicators stand out. These are products whose synthesis and manufacture have a high cost, and their implementation in jobs that involve monitoring the acidity or basicity of a system is simpler and faster (Soltan and Sirry, 2002; Sabnis, 2007; Singh *et al.*, 2011). However, synthetic indicators are not available to all interested persons in conducting studies and experiments. On the other hand, are natural indicators whose home collection is quite economical and uses fairly simple extraction methods (Esatbeyoglu *et al.*, 2012).

Curcumin, (1*E*, 6*E*) -1,7-bis (4-hydroxy-3-methoxy phenyl) -1,6-heptadiene-3,5-dione (C₂₁H₂₀O₆), is a natural dye from turmeric (*Curcuma longa*), a herbaceous plant of the family *Zingiberaceae* native to southwestern India. *Curcuma longa* is used as a spice in curry and as coloring agent in yellow mustards, cosmetics,

pharmaceuticals, and hair dyes (Ammon and Wahl, 1991; Toda *et al.*, 1985; Surh, 2003). It has attracted interest because of its antioxidant, anti-inflammatory, and potential anti-cancer activities (Aggarwal *et al.*, 2003; Anand *et al.*, 2007; Chattopadhyay *et al.*, 2004; Mishra and Palanivelu, 2008; Sharma *et al.*, 2005). Besides, curcumin has also been found to bind to β -amyloid proteins in models of Alzheimer's disease (Yang *et al.*, 2005).

This dye has an intense yellow color and is extracted from turmeric roots and stems (Wickenberg *et al.*, 2010). There are at least two forms of curcumin, both tautomers: the keto form and the enol form. The keto form is in liquid form, while the enol form is solid (Wickenberg *et al.*, 2010). Both tautomeric forms of curcumin are shown in Figure 1. This molecule can lose both hydrogens from either of the two -OH groups and the hydrogen located between the two ketones in a basic medium. Loss of any of these hydrogens occurs at pH 8-9 (Heredia, 2006).

The active ingredient in turmeric is the

polyphenol curcumin (responsible for its yellow color), also known as C.I. 75300, or natural yellow 3 (Breedlove, 1995; Carvalho *et al.*, 2002; Priyadarsini, 2014). It is used in food as a yellow dye (E-100i) or a flavoring agent, although it is also marketed for boron detection and as a pH indicator (Wickenberg *et al.*, 2010). Among the main physicochemical characteristics that curcumin presents are: an orange-yellowish color, specific gravity: 0.935, the melting point for the keto form: 183 °C. However, the boiling point for the enol form: decomposes, and density 0.93 g/mL. (Hatcher *et al.*, 2008) Curcumin has solubilities in ethanol, chloroform, acetone, glacial acetic acid, alkalis, and ether (Rios *et al.*, 2009). On the other hand, some researches have focused on curcumin, as acid-base indicator (Ma *et al.*, 2017). This dye is a lipophilic phytochemical that has been found to possess pH-dependent solubility (Wang *et al.*, 2019).

This work presents the extraction and purification of curcumin from the rhizomes of *Curcuma Longa* cultivated in Mérida, Venezuela. It is characterized by IR and UV-vis spectroscopic methods. The acid-base indicator character of curcumin is analyzed through the variation of the wavelength (λ) as a function of pH.

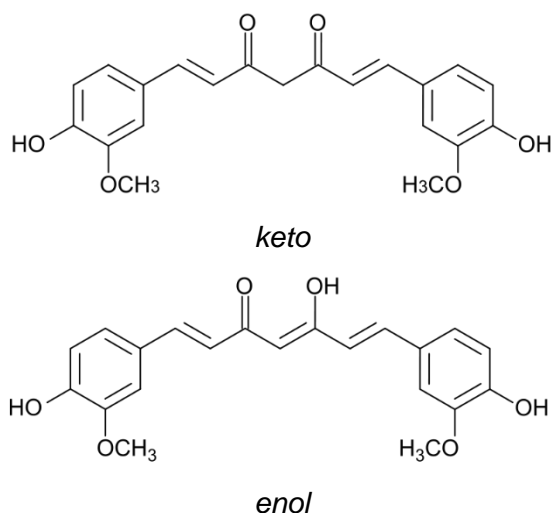


Figure 1. Keto and enol structure of curcumin

2. MATERIALS AND METHODS

2.1. Obtaining the turmeric rhizomes

The turmeric rhizomes of orange, fleshy, ovate, and piriform color come from the town of Lagunillas, Sucre Municipality of the Mérida state; This population is located at 1070 meters above sea level with an average annual temperature of 23 °C. A total of 1 Kg of turmeric rhizomes were collected for the study.

2.2. Preparation of the turmeric pulp

The rhizomes were subjected to washing, water and saturated NaCl solution, and chipping process, cutting them into small parts and placing them in a plastic container; the weight of the wet turmeric pulp was 717.0 g (Analytical Balance \pm 0.001g, model: AR3130). It was then placed in an oven at 65 °C (Brand: JP Selecta) for 4 hours, obtaining a final dry weight of 192.0 g. The dried turmeric was ground employing a grain mill (Corona), and 10 g was taken that was then passed through a degreasing process using three 20 mL aliquots of hexane (Fisher Scientific 99.8%) for 8 hours.

2.3. Coloring extraction

The extraction process was carried out for 7 hours using a Soxhlet extraction equipment (Brand: Klax.sa) of 250 mL capacity and 125 mL of ethanol (Reiedel de Haen, 95%) solvent. The orange extract was separated from the solvent in a rotary evaporator (Brand: Heidolph) at 60 °C, for 48 h.

2.4 Separation and purification of curcumin

It was performed by column chromatography, using a vacuum system (brand: Kimax) using Chloroform: Hexane 3:2 as eluent. The column was prepared in a 50 mL capacity burette (brand: Kimax \pm 0.1mL) with WN-3 type aluminum oxide: neutral (Sigma-Aldrich). The head of the column was made with 0.5 g of the extract in 2.5 g of alumina. The purity of the separated curcumin was checked by thin-layer chromatography (TLC).

2.5. Colorimetric pH scale

250 mL of sodium hydroxide (Reidel de Haen 99%) 0.105 M were prepared to weigh 1.05 g of the base by dissolving them in distilled water ASTM D1193 (ASTM, 2011). On the other hand, 250 mL of 0.1 M hydrochloric acid (Riedel de Haen 37%) was prepared, dissolving 2.1 mL of it in distilled water ASTM D1193 (ASTM, 2011). The indicator solution was prepared by weighing 7.0 mg of the purified curcumin in 15 mL of 95% ethanol. The proportions of the prepared solutions are shown in Table 6. A pH meter (Model Crison pH Meter Basic 20°C UPV) was used, and the results are shown in Table 6.

2.6. Characterization and quantification

FTIR spectroscopy: a Perkin-Elmer RX1 spectrometer, model 1605, was used for spectroscopic analysis using KBr pellets. UV-Vis

Absorption Spectroscopy: the solution was prepared in absolute ethanol (Riedel de Haen, reagent grade), scanned from 800 to 200 nm in a Shimadzu UV-mini 1240 spectrophotometer in a 1 cm quartz cell.

3. RESULTS AND DISCUSSION:

Pure curcumin was obtained by slow evaporation of the solvent, being a rather viscous and oily yellow liquid (Figures 2a and 2b), corresponding to the enol form of the compound of interest. Among the most important characteristic that could be obtained is that, as reported in previous studies, curcumin presented a decomposition reaction at temperatures greater than 80 °C, as well as a color change in basic medium to intense red. The amount of curcumin obtained is expressed in Table 1.

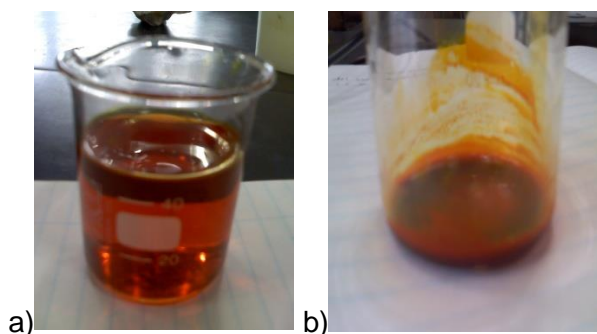


Figure 2. Photograph of the turmeric extract, (a) dissolution in 95% ethanol. (b) concentrated extract

Through the analyzes carried out on the substrate of the turmeric rhizomes, the percentage of humidity of these was determined compared to previous studies carried out in Quindio, Colombia, (Rios *et al.*, 2009) in which the variation of results based on the difference in altitude, soil characteristics of cultivation and climatological characteristics of each region that consequently alter the chemical compositions in the rhizomes shown in Table 2.

A thin layer chromatography (TLC) was performed of the extract obtained from the turmeric pigments, from the Soxhlet extraction in 95% ethanol, using chloroform as eluent observing three separate spots (Figure 3a); Knowing that the curcumin molecule has a low polarity, it could be assumed that it is the signal with the highest R_f. However, it was not possible to compare it with a standard sample as a reference.

The mixture of pigments contained in the extract was separated by column chromatography

(Figure 3b). There was a good separation of the pigments, the yellow pigment corresponding to curcumin coming out first (Figure 3c), showing high purity. This being verified by a thin layer chromatography whose R_f value resulted in 0.85 (Table 3).

The yellow fraction could not be recrystallized but, by slow evaporation of the solvent, a rather viscous and oily yellow liquid was obtained (Figure 3c), corresponding to the enol form of curcumin. Among the most important characteristic that could be obtained is that, as reported in previous studies, curcumin presented a decomposition reaction at temperatures greater than 80°C, as well as a color change in basic medium to intense red. The amount of curcumin obtained is expressed in Table 4.

3.1. Spectroscopic analysis

The UV-vis spectrum of curcumin in the enol form presented a maximum wavelength λ_{max} in absolute EtOH equal to 426.0 nm with an absorbance of 0.236 (dimensionless) (Figure 4). It was caused by the conjugation of the pi bonds as chromophores in the molecule. This type of transition of medium energy involved for this wavelength is $\pi \rightarrow \pi^*$, observing the yellow color as a complementary color, the wavelength is within the range of 420.0-430.0 nm of the chromatic circle. Likewise, the displacement value of λ_{max} agrees with the data reported for curcumin (Rios *et al.*, 2009).

On the other hand, it can be observed that in an aqueous medium, the solution with the curcumin indicator turns bright yellow, occurring transitions of the type $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$. For this spectrum, there is a wavelength λ_{max} equal to 426.0 nm and absorbance of 0.2332, seeing yellow as a complementary color (Figure 5). Something similar happens with the spectrum in an acid medium (Figure 6), with the difference of a slight bathochromic shift in wavelength at 432.0 nm and an increase in absorbance at 0.7028, known as hyperchromic shift because, in the solution in Acidic medium, turbidity is formed which causes the visible radiation that crosses the cell or optical path to be absorbed with greater intensity. In this way, the species that predominate in the aqueous medium is the keto-enolic form, according to the reaction shown in Figure 7.

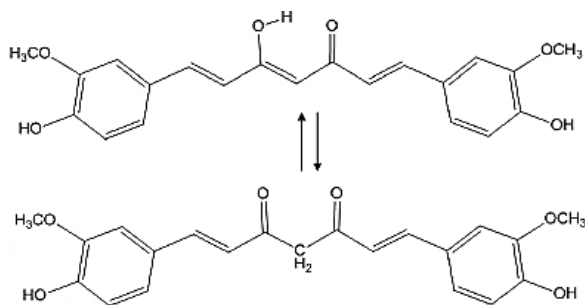


Figure 7. Keto-enol form of curcumin in the aqueous medium

Table 5 expresses the wavelength variation λ_{max} concerning pH on the colorimetric scale.

However, in a basic medium, there is a wavelength λ_{max} whose value is 461.0 nm, bathochromic shift towards the lower energy red and absorbance of 0.4070, observing the red color as a complementary color with transitions of the type $\pi \rightarrow \pi^*$ (Figure 9), predominating the molecular species shown below in Figure 8. It should be noted that these values agree very well with the values calculated theoretically for the transitions $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ of 483 and 343 nm, respectively (Rios *et al.*, 2009).

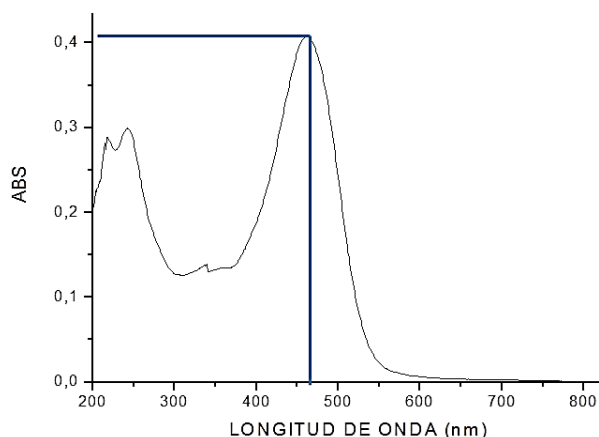


Figure 9. UV-vis spectrum of curcumin in basic medium

Concerning the characterization by FTIR spectroscopy of the purified curcumin (Figure 10), the main bands of the functional groups that coincide with the structure of curcumin in enol form are observed, identifying the strong tension signal OH at 3432 cm^{-1} , the voltage signal of the C=O link at 1650 cm^{-1} of medium intensity. This unusual intensity of this band is due to the delocalization of charge motivated by keto-enolic tautomerism that the curcumin molecule presents (Figure 1). Finally, bands of weak intensity are also observed between 2000 and 1900 cm^{-1} corresponding to conjugated C=C double bonds in the molecular skeleton.

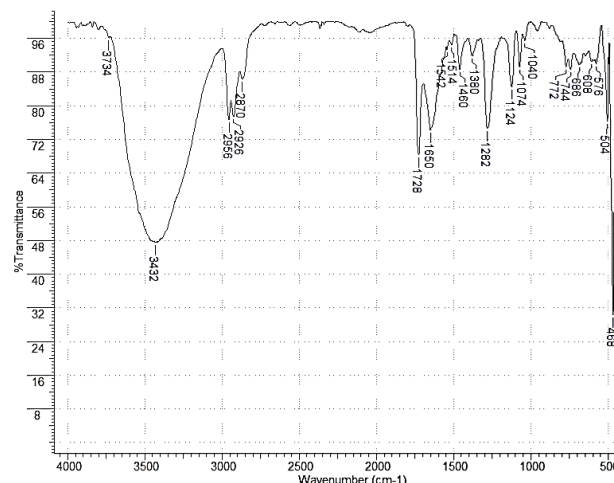


Figure 10. FTIR spectrum for purified curcumin in liquid (enol form)

The colorimetric pH scale was carried out to know the pH range of the change, being useful between pH 6-8. Table 6 shows the proportions of the solutions of the colorimetric scale with their pH value.

4. CONCLUSIONS:

It was possible to extract curcumin from the turmeric rhizomes through a simple method with a yield close to that reported in the bibliography and of high purity, verified by spectroscopic and chromatographic methods. Depending on the region where the crop is located, the concentration of curcumin can vary as well as the moisture content. This dye was useful as an acid-base indicator in strong acid-strong base volumes and did not require large amounts of it as it has high sensitivity summarize the data discussed in the Results and Discussion showing the relevance of the work and how different it is from others researches. Also, point out the benefits and improvements that can be observed to develop new science standards that can change something in the related field.

5. ACKNOWLEDGMENTS

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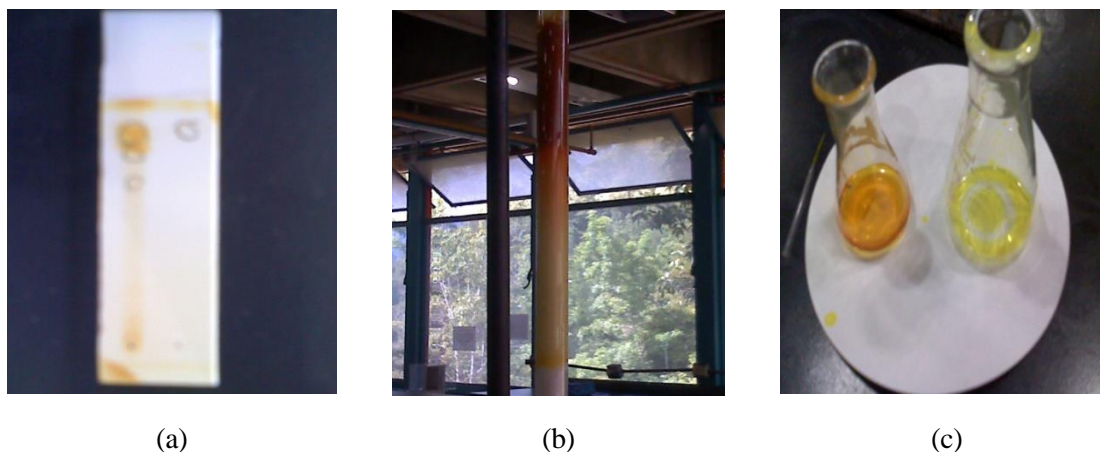


Figure 3. Photographs of separation and chromatographic purification. (a) Thin-layer chromatography of pure curcumin (application 2). (b) Separation of pigments in column (c) Pigments obtained

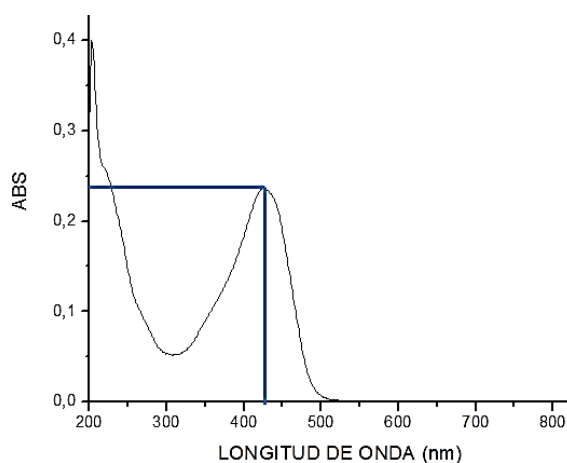


Figure 4. UV-Vis spectrum of curcumin in absolute ethanol

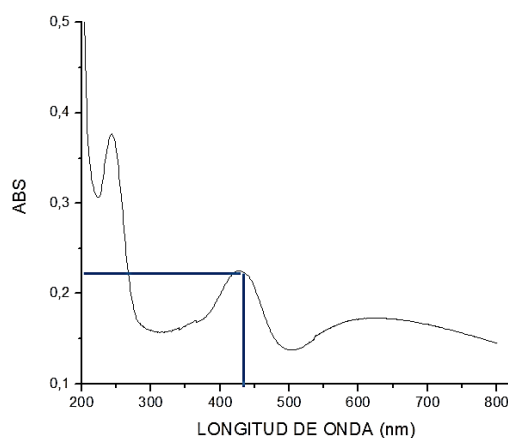


Figure 5. UV-vis spectrum of curcumin in the aqueous medium

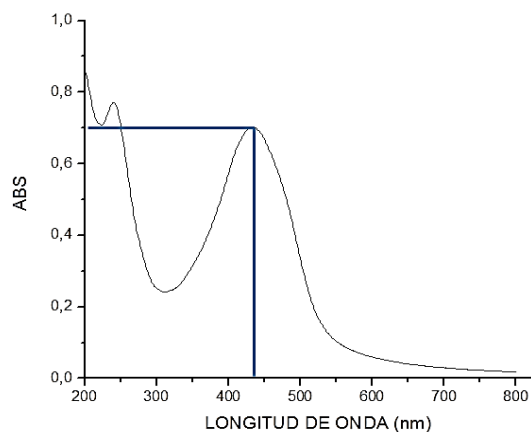


Figure 6. UV-vis spectrum of curcumin in acid medium

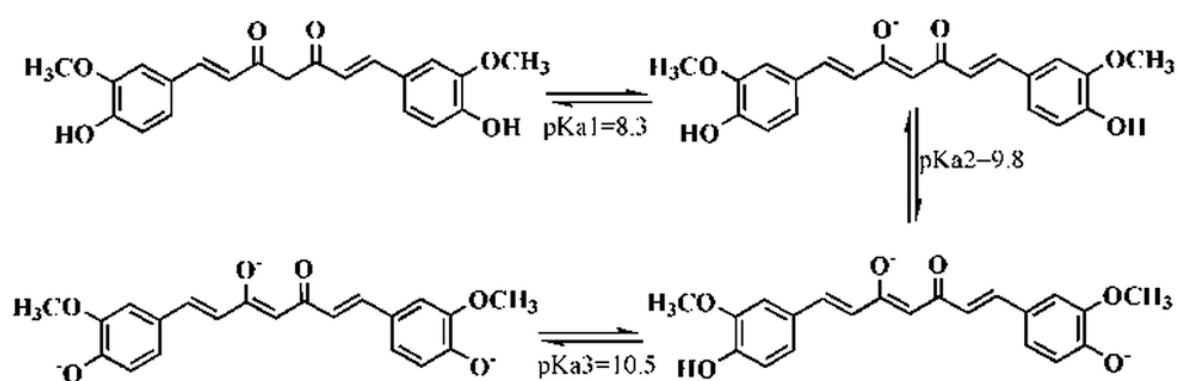


Figure 8. Mechanism of deprotonation of curcumin in basic medium

Table 1. The average amount of extract and curcumin obtained during the extraction processes with Soxhlet (ethanol).

m (flour subjected to extraction) (g)	m (extract) (g)	extraction yield (%)	m (curcumin) (g)	curcumin yield (%)
9.8 ± 1.6	0.9 ± 0.1	8.9 ± 0.7	0.030 ± 0.006	3.4 ± 0.3

Table 2. Determination of relative humidity and percentage of the fatty extract of the turmeric rhizomes studied and compared with a crop from another region

Region	Altitude (masl)	Annual average temperature (°C)	Initial substrate mass (g)	Dry substrate mass (g)	% of relative humidity (g)	% of fatty extract
Mérida-Venezuela	1070	21-23	716.975	192.035	72.9 ± 1.3	6.8 ± 0.8
Quindío-Colombia	900-4750	18-21	NR	NR	84.7 ± 3.0	0.05 ± 0.02

Table 3. Thin-layer chromatography performed on the extract of turmeric pigments

Aplication	signal	signal height (cm)	solvent front (cm)	Rf
1	A	2.3	4.0	0.57
	B	3.0		0.75
	C	3.4		0.85

Table 4. Amount of extract and curcumin obtained during the extraction processes with Soxhlet (ethanol)

Experiment	m _{flour} subjected to extraction (g)	m _{extract} (g)	extractio n Yield (%)*	m _{Curcumin} (g)	curcumin Yield (%)**
Exp. 1	10.0	1.1	10.7	0.039	3.7
Exp. 2	10.0	0.8	8.4	0.026	3.1
Exp. 3	9.3	0.7	7.6	0.024	3.5
Average	9.8 ± 1,6	0.9 ± 0.1	8.9 ± 0.7	0.030 ± 0,006	3.4 ± 0.3

* The extraction yield was determined with the mass of the oily extract of the pigment mixture concerning the initial mass of the dry turmeric. ** The yield was calculated considering the mass of the oil obtained concerning the concentrated extract of the pigment mixture.

Table 5. Variation of wavelength and absorbance with the change of pH

Medium	wavelength λ_{\max} (nm)	Absorbance	Color	Medium
aqueous	631.0	0.1732	Yellow	aqueous
	426.0	0.2253	Yellow	
	243.0	0.3765	Yellow	
acid	432.0	0.7028	Yellow	acid
	240.0	0.7708	Yellow	
	461.0	0.4070	Red	
basic	243.0	0.2996	Red	basic
	219.0	0.2883	Red	
	631.0	0.1732	Yellow	
aqueous	426.0	0.2253	Yellow	aqueous
	243.0	0.3765	Yellow	
			Yellow	

Table 6. Variation of pH as a function of the variation of the acid and base proportions of the colorimetric scale

Solution	V _{NaOH 0.1M} (mL)	V _{HCl 0.1M} (mL)	number of indicator drops	pH	Solution color
1	0	10	10	1.07	Yellow
2	1	9	10	1.78	Yellow
3	2	8	10	2.01	Yellow
4	3	7	10	2.66	Yellow
5	4	6	10	3.17	Yellow
6	5	5	10	6.52	Orange
7	6	4	10	10.66	Red
8	7	3	10	11.40	Red
9	8	2	10	11.54	Red
10	9	1	10	11.88	Red
11	10	0	10	12.59	Red

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ABSTRACT

The ore beneficiation process uses little technology, but mining companies have high economic performance. As an energy source, firewood plays a fundamental role due to its simple storage, low cost, great availability of forests, and lack of processing. Thus, the present work aims to analyze the consumption of firewood as fuel and possible improvements in the process for the production of lime in terms of harnessing raw materials and costs. Calcium oxide is obtained from the thermal decomposition of calcium and magnesium carbonates obtained from dolomitic deposits of limestone (CaCO_3 : CaMgCO_3). After CaO extraction, it is subjected to a calcination process, removing carbon dioxide (CO_2) in ovens that work at temperatures between 900 and 1200 °C. The source of energy applied to the calcination furnaces in the analyzed area is wood. The wood has a calorific value between 2,250 and 2,700 Kcal/Kg, but the moisture content responsible for the thermal variation must be considered. The firewood burning process was carried out in a ravine type oven where the temperature at which operators are exposed to heat was evaluated. The results indicated that the cooking time dropped by 20% as the amount of wood is fed into the oven. This increase represents a significant gain in lime production, thus leading to a higher profit for the company.

Keywords: *Calcination, Limestone, Firewood, Dolomite, Biomass.*

1. INTRODUCTION

Among the most different products derived from mineral sources, lime, also called quicklime, virgin lime, or calcium oxide (CaO), is considered the most important in the market in terms of applicability and volume consumed (Dietzen *et al.*, 2018; De Souza and Bragança, 2017; Barbosa *et al.*, 2016; Nouri and Ebrahim, 2016; Van Straaten, 2006; Wallmann and Aloisi, 2012; West and McBride, 2005). Calcium oxide is obtained from the thermal decomposition of calcium and magnesium carbonates obtained from dolomitic limestone deposits (CaCO_3 : CaMgCO_3) (Palma *et al.*, 2018; Khadra *et al.*, 2017; Chen *et al.*, 2016; Gomes *et al.*, 2016; Koenig and Liu, 2002). By definition, dolomitic limestone is a mineral that has limestone with the highest concentration of calcium and magnesium oxide (Erans *et al.*, 2019). After CaO extraction, it undergoes a calcination process, removing carbon dioxide (CO_2) in furnaces that work at

temperatures between 900 and 1200 °C (Yuan *et al.*, 2018; Asano *et al.*, 2014; Stanmore and Gilot, 2005; Klunk *et al.*, 2020a; 2020b). In the current economic scenario, the rising price of energy resources has led many companies to seek efficient tools for their processes (Erans *et al.*, 2019). Plant biomass is widely used for energy production (Cataluña *et al.*, 2018; Caetano and Silva, 2017; Cataluña *et al.*, 2017; Nascimento *et al.*, 2017; Rodrigues *et al.*, 2016; Safari and Zeynizadeh, 2015; Ishmayana *et al.*, 2015).

The synthesis of nanopore materials such as molecular sieves (zeolites) has been gaining worldwide importance to increase the efficiency in obtaining high value-added products (Sadjadi and Reza Naimi-Jamal, 2019a, 2019b; Moreira *et al.*, 2018; Moradi *et al.*, 2014). In developed countries, advanced technologies allow a higher yield in CaO (Ferreiro *et al.*, 2019; Dai *et al.*, 2016). The energy source applied to calcination furnaces in the analyzed area is wood (Glasenapp *et al.*, 2019; Aguilar *et al.*, 2016; Arabatzis and Malesios, 2011). Wood has a

calorific value between 2.250 to 2.700 Kcal/Kg, but the moisture content that is responsible for a thermal variation must be considered (Aguilar *et al.*, 2016; Song *et al.*, 2012; Couture *et al.*, 2012). As a result, the burning process can cause high raw material (wood) expenditures, excessive heat emitted by fire, and environmental pollution (Pereira *et al.*, 2018; Reguieg *et al.*, 2017). The use of wood for energy purposes has aroused the interest of the industrial sector because it is a renewable resource and low production cost (Caetano *et al.*, 2015a; 2015b; 2015c). The main species cultivated in Brazil to obtain energy belong to the genus *Eucalyptus* sp (Pimenta *et al.*, 2018; Flores *et al.*, 2016; Barbosa *et al.*, 2016). Knowing the importance of wood, it is necessary to know its characteristics, having as its fundamental principle its origin (Filomeno *et al.*, 2016; De Almeida *et al.*, 2017; Da Silva *et al.*, 2016; Mossi *et al.*, 2010).

Oven operators are exposed to high temperatures daily on their workday. Under prolonged conditions, this can lead to damage to health and loss of work performance, thus increasing the likelihood of accidents. For this reason, the monitoring of the temperature that the operators are exposed to is essential so that it is adequate for the execution of the activities. The company considers working conditions essential to increase production capacity and gain production. A field study verified the necessity of the company, located in the city of Caçapava do Sul, to improve the fuel efficiency (wood) used in the lime kiln. The amount of dolomitic limestone, firewood for energy generation, and lime produced seek to propose improvements in the process to obtain the final product (CaO), offering a higher yield. This paper aims to analyze the fuel consumption (wood), the raw material (limestone) for lime production in a calcining furnace in terms of raw material utilization and costs, as well as a temperature monitoring at which the operator was exposed. Quantitative analyzes of radiated heat were performed using a thermal stress meter (Tgd 400 Instrutherm). Thus, the results were compared with the tolerance limits established by Regulatory Standard NR 15.

The general parameters used by this study are: i) to analyze the consumption of raw material; ii) carry out an economic assessment of the costs involved in the process; iii) propose changes in the process to optimize production costs and yield and iv) analyze the occupational risks that employees are exposed to. Lime is a fundamental product of the economic activity of most countries and is used worldwide in the most

varied sectors (construction, agriculture, food industry, among others) (Moradi *et al.*, 2014; Dai *et al.*, 2016). In this context, obtaining calcium oxide is expensive, and the main challenge for companies is to reduce the consumption of the fuel used (wood), thus optimizing production (Aguilar *et al.*, 2016; Arabatzis and Malesios, 2011).

The virgin lime manufacturing process takes into account the use of different types of fuels (Yuan *et al.*, 2018; Asano *et al.*, 2014). Fuel sources have an influence on product quality and manufacturing costs, so the choice of fuel must be carefully delineated. In quantitative terms, a calcining furnace uses on average 1,800 to 2,500 tonnes of firewood per month. The process of obtaining calcium oxide implies the emission of large amounts of carbon dioxide (1 ton of lime generates 1.2 tons of CO₂), mainly responsible for climate change (Sadjadi, 2019; Sadi-Nezhad, 2019; De Aguiar *et al.*, 2017; Soares *et al.*, 2016). These CO₂ emissions can be predicted by geochemical modeling (Bigarani *et al.*, 2016). Another important factor to take into account is changes in the landscape because of limestone mining and detachment of particulate matter (Lawer *et al.*, 2019; McPhail, 2009; Brunnschweiler and Bulte, 2008).

Lime companies increasingly need to ensure the quality of the end product, improve production processes, leading to sustainability and reduce operating costs to stay in such a competitive market (Pimenta *et al.*, 2018; Flores *et al.*, 2016). In this context, fuels represent today one of the largest costs in lime companies that have ravine ovens (Rodrigues, 2015). Concern about the health and safety of workers exposed to lime production and processing reflected positively on the industrial sector. In this regard, labor laws ensure that workers must be in a protected environment by adopting appropriate measures for their comfort and well-being.

2. MATERIALS AND METHODS

Provide sufficient details to permit repetition of the experimental work. The technical description of methods should be given when such methods are new.

2.1. Location and characterization of the study area

The studies of this work were performed in a company from Caçapava do Sul city, located in the Rio Grande do Sul/Brazil. Its territory is

located in the so-called Campaign Zone, with large deposits of copper, lime and kaolin ores. In its topographic configuration, we can see grand fields and mountains, with dark lands and silica-rich soil (Horn *et al.*, 2017). Its economy is fundamentally supported by the agriculture, livestock, and mining sectors, where local limestone production represents more than 80% of what is produced in the Rio Grande do Sul. The extraction of minerals in the company, basically dolomitic limestone, is aimed at the production of mortar and lime, used in areas such as construction and agriculture to correct soil acidity, with the aid of a forno de barranco, named after being supported by a slope, with dimensions of four to five meters in height and conical trunk shape. The fuel (wood) can be added directly to the load or burned inside furnaces. In these configurations, the consumption of wood for energy purposes is high (Guimarães, 1998).

The ravine type analyzed in this work uses black wattle firewood (*Acacia mearnsii*) and eucalyptus (*eucalyptus* sp) as fuel. The calorific value in the dry state is 4,800.54 Kcal/kg, and in the wet state 2,500.61 Kcal/kg (Caetano *et al.*, 2018). It was verified the inexistence of moisture control in the wood leading to higher or lower levels of carbon dioxide emissions. This impairs the temperature control and, consequently, the calcination process. Without this control, the consequence is high costs, high raw material consumption, and loss of process efficiency.

The company has 258 employees, 14 of which directly involved in the lime calcination process. In this place, there are 4 ravine ovens with a capacity of 80 tons/day of dolomitic limestone with a production of 47 tons of lime. For each ton of lime produced, 1.3 m³ of firewood is required. For the calcination process, temperatures in the range of 900 to 1,200 °C must be reached. The company operates 24 hours a day, seven days a week, based on foreign market demands. Refueling is done every 2.4 hours. As the dolomitic limestone goes down inside the furnace, the heat exchange by heating of the hot gases occurs until reaching a necessary temperature level for the combustion, initiating the calcination process. In the calcination process, the mineral (dolomitic limestone) is deposited in the oven where it remains for around 24 hours, cooking at initial temperatures of 300 to 500 °C until reaching higher temperature levels (900 °C). Product cooling is given in the lower chamber. The material is removed from the bottom of the

cooling chamber, where it is waiting to be loaded for the next production step. The calcination process is completed by cooling and transported in wagons to a deposit, where it will proceed to the milling process. The current consumption of firewood is 1,936.06 m³/month, being black wattle 922 m³/month and eucalyptus 1,014.91 m³/month. The cost of black wattle is R\$52.00 per m³, and eucalyptus of R\$53.00 per m³. The energy was calculated by active power during the operating time.

Proper calcination will depend on dolomitic limestone, furnace operating conditions, and the quality of the raw material. However, the lack of knowledge of the relationship of these factors in the quality and productivity of virgin lime makes the operation of the ovens depend exclusively on the operator's experience. Some of these factors that may interfere with calcination, and whose careful handling of the furnaces allow its control, are the retraction of dolomitic limestone, recharge, and steam effect (Guimarães, 1998).

2.2. Firewood Consumption

The fuel consumption of the company's ovens was analyzed based on the wood used for each batch. The study performed a continuous follow-up of 10 batches (daily use). Whereas each batch has 2 tons of dolomitic limestone and is made around 10 daily. Characteristics of the calcination process, taking into account the company's theoretical data:

- (i) kiln capacity: 1.7 m³ firewood per tonne of virgin lime;
- (ii) Consumption of firewood per batch: 10.4 m³;
- (iii) Firewood consumption/tonne of lime: 1.3 m³ for 1 tonne of lime;
- (iv) Time of the batch: 24 hours.

According to Silva (2009), ravine ovens have high fuel consumption (on average, 220 kg of fuel oil or 1.7 m³ of firewood per ton of virgin lime). Firewood consumption in this process is 13.6 m³ per batch, an increase of 30% when compared to the theoretical data of the companies. This discrepancy lies in the fact that there is no control of wood moisture parameters and stoichiometric weighing of the raw material.

The conversion rate in carbonate calcination is adjusted by providing heat for decomposition, taking into account particles larger than 40 mm. In this way, calcination is governed by diffusion law in controlled reactions: the decomposition time is proportional to the square of the particle diameter, so the conversion

rate is inversely proportional to the particle diameter. According to Erans *et al.* (2019), the evaluation of calcination kinetics is relatively complex due to factors such as:

- i) CO₂ concentration (slows the reaction);
- ii) particle size (limiting factor in energy transfer);
- iii) catalytic inhibition (presence of impurities).

The carbonate calcination rate becomes high when the temperature is reached, where the partial equilibrium pressure of CO₂ or water becomes equal to the total reactor pressure. This temperature is called the decomposition or calcination temperature. The reaction rates and reactivity of virgin lime are directly linked to the temperature at which lime was calcined. The higher the process temperature, the less the calcination time, resulting in lower production costs.

2.3. Cost analysis involved

It was verified in the analysis, and it was verified the fixed costs (firewood in the energy generation) to minimize the losses in the calcination process. This parameter is closely linked to the company's profit. Once we can reduce the quantities of this fuel, we will have a significant profit from the company.

3. RESULTS AND DISCUSSION

3.1. Firewood Consumption

According to the company's operational survey, there are 10 batches per day, with 2 tons of dolomitic limestone each, totaling 8 tons per batch of raw material. Considering that the process uses 1.3 m³ of firewood for each ton of lime, at the end of the process, we have a total volume of 1,936.54 m³ of burnt wood for power generation.

According to Lins (2007), 1.7 tons of limestone is required for each ton of virgin lime. Therefore, increasing the amount of firewood reduces cooking time by 20%. This way, it can increase the amount of matter per batch. The higher the heat flux transmitted to dolomitic limestone, the shorter the cooking time. In table 1, find the actual scenario of the company.

In an ideal operating condition, if the company used the full capacity of the oven (1.7 m³ of firewood), the calcination time would decrease by 2 hours because the higher the furnace temperature, the higher the production of lime oxide. Table 2 shows these new values.

Using the ideal scenario, the production would gain 93 tons of lime produced per month, in a year would increase by 1,116 tons. If the company had greater control of the data, other types of process optimization could occur. According to Viana *et al.* (2012), the total loss basically consists of:

- i) heat transfer losses - the heat absorbed by the oven walls, ceiling, and the threshold is released by radiation and convection.
- ii) leakage losses in the furnace structure - which usually operate at higher than atmospheric pressure, and heat losses through the door when it is opened.
- iii) chimney losses - are the losses associated with the dry gases formed in the combustion and the losses near the vapor present in the chimney.

3.2. Cost analysis

The company buys firewood from six suppliers in the region, with a black wattle price of R\$ 52.00 and eucalyptus R\$53.00/m³, respectively. The current consumption of firewood is 1,936 m³/month, with acacia 922 m³/month and eucalyptus 1,014 m³/month, totaling a monthly expense of R\$101,686.00. In table 3, find the fixed costs related to the oven.

Corrective maintenance is carried out, but usually due to the belts and trolleys involved in the furnace, but in the furnace itself, maintenance is performed every six years, with the replacement of some furnace components (refractory and revocations), having a hand cost of work and raw material of R\$10,000.00.

Performing a profitable cost analysis, according to market research, we have according to table 4. Moisture control of firewood is recommended in order to reduce calcination time, reducing waste and waste, and optimizing the process as a whole.

3.3. Worker welfare analysis directly involved in the process

Temperature measurements were performed near the oven and where workers perform their duties. Climatic factors of the region contribute to the thermal overload of the operators, where they intensify in the summer periods. Another factor contributing to thermal overload is the opening of the furnace for cooling. In this process, heat conduction intensifies, leaving the workplace with temperatures higher than those determined by law. The results of temperature measurements are shown in Table

5. According to the results, temperature measurements in the various oven zones tell us that at the inlet of the oven, there is a warm environment ranging from 38 to 40 °C. This temperature directly affects the operator who is exposed to this location during his workday. This is due to the internal temperature of the oven (1000 °C on average), as thermal conduction to the outside occurs when measured at the top outside of the oven (300 °C on average).

According to NR 15, operator activity under these conditions is arduous and requires 15-minute breaks for every 45 minutes worked. Even if the operator is using individual safety equipment, exposure to high temperatures can affect their biological system, causing some health damage.

4. CONCLUSIONS

With the results obtained in this work, it was possible to characterize the fuel (wood) of the calcination furnace as well as the costs involved in the process. In this analysis, it is found that increasing the fuel capacity leads to gain in virgin lime production. The amount of wood used to burn dolomitic limestone has a significant impact on the company's final cost. Wood moisture measurements are a key parameter to increase the yield of calcination reactions. Regarding the exposure of workers to high temperatures, we can conclude that they are in an unhealthy work situation. These operators are above tolerance limits when compared to NR-15. The results indicate that the work environment in the company's furnace provides an exposure situation above the recommended by legislation. According to the company's board, these results were of fundamental importance, leading to corrective and preventive actions towards its operators. Thus we can conclude that the real and significant gain of the company is closely linked to the optimal working conditions of its workers and investments in new technologies, thus being competitive with the market and its competitors.

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Table 1. Actual business operating condition

Batch	Quantity of raw material	Time	Firewood used daily	Daily production
10 a day	80 tons a day	Every 2.5 hours the ovens are supplied, remaining for 24 hours	61.2 m ³	47 tons of lime

Table 2. Ideal scenario.

Batch	Quantity of raw material	Time	Firewood used daily	Daily production
11 a day	88 tons a day	Every 2 hours the ovens are supplied, remaining for 24 hours	58 m ³	47 tons of lime

Table 3. Fixed Costs for the company's oven

Firewood Acquisition	Maintenance	Employees	Total
R\$ 1,220.232/year	R\$ 10,000.00/year	R\$ 543,365.05/year	R\$ 1,773,597.05/year

Table 4. Production versus gain

Final product price of lime 0.02 tons	Values according to monthly production of 1.457 tons	Values according to monthly production of 1.550 tons	Earnings
R\$ 12.00	R\$ 874,200.00	R\$ 930,000.00	R\$ 55,800.00

Table 5. Temperature measurements taken within 5 days

Days	Initial temperature near oven inlet (°C)	Oven top outside temperature (°C)	Oven temperature (°C)	Oven physical space temperature (°C)
1	40	300	1030	9
2	39	240	1005	10
3	38	290	1000	9
4	40	285	1030	10
5	40	300	1000	12

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ABSTRACT

Sand casting is a viable waste as an alternative raw material, regarding a good solution in the production of concrete blocks. The use of sand casting to produce concrete blocks is performed by about 80% of the productive capacity of the company considered in this study. Thus, this work aims to make an improvement by applying fuzzy logic. The methodology was applied from the formulation of ranking criteria, which improved the production process. Therefore, after the implementation of the established criteria, was achieved an improvement of about 10%. Thus, the results achieved can help companies, generating alternatives with more excellent reliability and assertiveness. Moreover, Fuzzy is a methodology that provides strong support for decision making, supporting subjective and intuitive criteria.

Keywords: Fuzzy Logic, Production Management, Concrete Blocks, Casting Sand.

1. INTRODUCTION:

The economic empowerment of the whole world is based on a balanced economic, political, social, cultural, and environmental model (Cataluña *et al.*, 2018; Zirham and Palomba, 2016). All generations can follow on radio, television, and newspapers, seeking the commitment of all so that they meet the needs of current generations without compromising the ability of future generations (Best, 2017; Cataluña *et al.*, 2017).

With the accelerated development process and the dynamization of the market, organizations are obliged to look for ways to improve their operations, thus optimizing the final results obtained. Besides, another relevant factor is companies considering the consequences of their actions, thus denoting concern for the environment, adopting policies and measures that lead to a sustainable workforce (Venturini *et al.*, 2018; Caetano *et al.*, 2018).

However, this research seeks to show the large amount of "foundry sand" waste produced and its reuse as an alternative raw material to replace normal sand in the production of concrete blocks (Sua-iam *et al.*, 2019). For example, it is

shown in the results of a study that concrete workability decreases as the amount of foundry sand increases, which was attributed to the larger surface area of this sand compared to the aggregate used (Prasad *et al.*, 2018). In this case, in addition to the utilization, there is greater resistance in the product due to the components added to the sand in the matrices (Torres *et al.*, 2017).

In this case, in a constant transformation market that is continuously emerging new goals and objectives to be achieved, managers require a lot of attention to find better tools and solutions to manage the production (Gerônimo *et al.*, 2017; Caetano *et al.*, 2017). Moreover, production management models have traditionally been composed of two dimensions: the technical dimension and the social dimension (Pérez-Barea *et al.*, 2018). The technological dimension refers to production, such as processes, activities, the physical arrangement of equipment, and the flow of material that results in goods and services (Zou *et al.*, 2019).

The other dimension relates to social and refers to the organization of work. In such a case, there are essential elements for a company to become competitive, such as strategy, product

development, logistics, production management, and manufacturing technology, where all these elements must be integrated to optimize processes based on the market demand of the organization (Zhu *et al.*, 2019; Sutherland *et al.*, 2016). An example is a study of Pablos *et al.* (2012) who devised a methodology that establishes a waste encapsulation process using the solidification/stabilization technique in Portland cement matrices. This process makes the residues act as small and large aggregates in the concrete composition (Okoronkwo *et al.*, 2018).

Thus present the possibility of using this concrete in the manufacture of concrete pieces (blocks). In this context, industrial waste, where it is disposed of makes organizations pay a high price for it (He *et al.*, 2019; Caetano *et al.*, 2015). However, foundry sand is a viable reuse residue as an alternative raw material to replace normal sand, so it is an economically advantageous solution. (Matos *et al.*, 2019). The authors also suggested a methodology to reuse these solid wastes by testing the use of shingles incorporating foundry sand in different concentrations, in increasing order of 5%, where natural sand was replaced by foundry sand, where the result of the methodology was satisfactory for consumption. Current studies highlight the concern with the environmental sector, preserving natural resources and waste mainly in civil construction (Wahi *et al.*, 2016; Sieffert *et al.*, 2014).

Therefore, it is observed in research the approach of the use of foundry sand as a base component of mortars, concrete blocks, concrete pavements, and self-compacting concrete. (Kruger *et al.*, 2013). Consequently, this research seeks to apply fuzzy logic to perform optimization simulations of producing concrete blocks from foundry sand, aiming to enable managers alternatives that generate improvements to their products. In this scenario, the criteria to be evaluated are equipment, time, cost, and human resources involved in producing the blocks (Iqbal *et al.*, 2019). According to Prabakaran *et al.* (2018), investigating the process of reducing fertilizer consumption and improving crop productivity using fuzzy logic systems is relevant. Data were analyzed in MATLAB to establish feasibility rules for crop decision support systems.

This justifies the importance of using the fuzzy logic theory to achieve relevant results in the use of foundry sand to produce concrete blocks (Beheshti Aval *et al.*, 2017). Considering that there are several examples in the literature that were considered successful in research, the fuzzy logic was used. Thus, through this theory, it is possible

to understand the criteria so that they may contribute to the performance of the organization under study (Carbajal-Hernández *et al.*, 2012).

In an organization, the production process is inherent in the extraction, manufacture, distribution, consumption, recycling, and reuse of resources, as these relationships become paramount to optimize its performance (Klunk *et al.*, 2019a; Ruoso *et al.*, 2019). Also, production management can be defined as a function of planning management, organization, direction, coordination, and control of inputs and process elements and has the objective of manufacturing high value-added goods and services (Klunk *et al.*, 2019b). The operations of a production process are controlled in time and space and are assisted by suppliers and customers (Tezel *et al.*, 2016).

In the modern context of production management, organizations have large amounts of data sourced from past industrial processes due to current technology and the benchmark. However, large data volume makes it difficult to quickly convert data into action for the benefit of an organization, but this is extremely important so that process cycles become shorter (Fraga *et al.*, 2014; Klunk *et al.*, 2012). In this way, an organization's data analysis capabilities make it a competitive advantage, optimizing production management in decision making (Bumblauskas *et al.*, 2017). Thus, production management has traditionally been defined as the set of activities required to plan and control the manufacturing process. Regarding the theme, given the current market competitiveness, the challenges of companies is to innovate so that products are relevant items in the face of consumer market influence. Besides, innovation needs to be valued at long, medium, and short-term strategic planning levels, as it is a tool that impacts cost savings and increases quality (Gerônimo *et al.*, 2018).

Given this, the appropriate use of sustainable resources aims to bring economic benefits to production processes to reduce the impact on environmental interactions of resources beyond the limits (Bentes *et al.*, 2012). Thus, with the growing interest in preserving the environment, it is necessary to explore ways to transform, recycle, and reuse the industrial waste in building materials.

It is noted that the metallurgy industry generates waste that is no longer used throughout the production process, where the inefficient waste management can characterize an environmental problem (Iluțiu-Varvara *et al.*, 2017). Thus, one of the wastes generated by these

organizations is foundry sand, which is of uniform size and high quality and is fine sand useful for reuse (Sithole *et al.*, 2019). Moreover, over the years, this material has been disposed of in landfills. Still, the large volume currently generated costs have become a problem in the United States, which has about 3000 foundries, using 100 million tons of sand annually, and generating about 6 to 10 million tons of foundry waste (foundry sand) that is disposed of in landfills (Dolage *et al.*, 2013).

In Brazil, foundry sand is the industrial waste with the highest volume since each ton of molten metal generates another ton of sand to be disposed of. Foundry sand can be defined as a raw material based on other chemicals to make molds in the metalworking industries. In this context, it requires cultural and awareness changes, so research is found that refers to the successful use of industrial waste foundry in concrete blocks (Fernandes, 2004).

Thus, the challenge is to find sustainable solutions that lower the high costs of landfill disposal and generate a return for the metallurgy industry, as waste sand is a safe material for engineering applications. Thus, to assist in managing industrial waste production, fuzzy logic with triangular numbers was used to elucidate improvements in the production of concrete blocks using foundry sand (Matos *et al.*, 2019).

Fuzzy logic is widely used to solve decision-making problems (Iqbal *et al.*, 2019). Fuzzy Logic (also called multivalued logic) was introduced in 1930 by the Polish philosopher and logician Jan Lukasiewicz (Godo and Gottwald, 2015). Using terms like tall, old and hot, he proposed using a range of values [0,1], which would indicate the possibility for a statement to be true or false (Lim *et al.*, 2016). In 1937 the philosopher Max Black suggested the concept that continuity described degrees (Black, 1942).

Given this, this logic uses the modeling of systems with poorly defined boundaries; when working with complex processes, it is observed that several classes of objects do not have a well-filled belonging criterion, with inaccurate or approximate information. Thus, it comes from convergence to fill the lack of theories that use a usual mathematical treatment for linguistic variables. The concept is that the values are attributed by words or sentences in the natural or artificial language (Zadeh, 1996; Lima Junior and Carpinetti, 2015).

Thus, the Fuzzy approach is pertinent when using qualitative linguistic variables such as

low, medium, high, and probable, unlikely, interpreted by Fuzzy numbers and articulated by valid arithmetic. Consequently, it makes it possible to consider a more significant number of variables, allowing simulations of the human judgment process, thus facilitating the search for problem-solving and making the foundation of a knowledge base more straightforward. However, data processing speed is one of the reasons that hinder the application of logic in multiagent modeling outside the academic context. (Braga *et al.*, 2017).

It is noteworthy that within fuzzy sets, a particular variable can belong to more than one linguistic set, a fuzzy set where values from 0 to 1 are provided in each element, where it represents the degree to which it belongs to the inaccurate concept to the set. Fuzzy Consequently, the numbers are part of a Fuzzy inference group; the first step is to convert qualitative variables to fuzzy numbers having the name of fuzzyfication, followed by data processing, ie inference based on fuzzy rules, Finally, defuzzyfication consists of the information analysis stage of the fuzzy set (Moraes, 2014). Thus, the next topic characterizes the methodology outlined for this research, aiming to elucidate all the means employed to achieve the defined research objective.

2. MATERIALS AND METHODS:

A scientific study contributes to the obtaining of knowledge. Where the results found help decision-makers find the best alternatives to improve their organizations. The research on the approach can be pointed as qualitative and quantitative due to the methods employed. Regarding the central themes, the research is still classified as a case study because it sought to systematically describe the characteristics of the company and the application of fuzzy decision support logic to hierarchy for decision making (Vergara, 2002).

Based on this assumption, to define the scope of fuzzy methodology, sets, and logic aiming at the modeling and development of control systems, it will be evidenced only what is necessary to understand the basic fuzzy control theory (Qureshi *et al.*, 2018). Interpretations through fuzzy logic of a data structure is a natural and intuitively plausible way to formulate and solve various problems. In this sense, the methodology of the methodology, as the nature of the research, can be considered applied due to the practical answers obtained and implemented (Abaei *et al.*, 2018). Regarding the objectives is exploratory and descriptive, having the need to study in more

depth the concepts and characteristics of the problem. The approach is qualitative and quantitative, and it is a case study. This research was carried out in a concrete block industry, being an exploratory study, because it was sought to have more familiarity with the subject, where through observations, it was verified how was the productive process of the manufacture of concrete blocks from of foundry sand (Makul *et al.*, 2018).

The data were obtained through the interview and observation. This one can define each of the criteria, working with Cr1 in units, Cr2 in day, Cr3 in reais, and Cr4 in the number of employees. Thus, the company under study can be considered as a universe of all employees of the organization. We used the fuzzy logic for analysis purposes, with fuzzy triangular numbers, developed by Lofti Zadeh in 1965, with these fuzzy sets (Zadeh, 1996). Since then, other researchers began to use it; within this theory, it is understood that within each set, there are gaps or vague phenomena, serving such methodology to describe and verify them. Consequently, this research seeks to apply the logic fuzzy logic to perform simulations of optimization of the process of production of concrete blocks from foundry sand, thus enabling to define of better alternatives that generate improvements in products (Gent *et al.*, 2015).

3. RESULTS AND DISCUSSION:

3.1. Initial Diagnosis

Understudy, the organization has a corporate characteristic framed by the IRS as a Small Company - EPP, being a family structure industry. The company was opened in 2007, and is managed by two partners, and has four employees, and the organization operates directly in the field of block manufacturing.

Thus, the company manufactures pre-lage, cement, concrete, Roman tile, bricks of all types and sells building materials in general. However, the main product sold by the industry under study is concrete blocks made for masonry and paving, which has a production capacity of 90,000 pieces/month. However, the organization currently manufactures about 72,000 pieces/month of concrete blocks.

Also, the company makes available from a remote area of the city due to the sound emitted by the machinery for the manufacture of concrete products and parts. The site has a space for locating machines, equipment, warehouses, among others. Its facilities are housed in a pavilion

with about 800 m² with a total area of approximately 20,000 m², with a production cost of R\$ 59,200.00.

3.2. Analysis of the concrete blocks production process

In the initial stage, the foundry sand is received and then conveyed to the hoods by conveyor belts to be weighed separately. After this, the residue is mixed in a mixer where additives and cement are added together with the foundry sand, thus generating base material for the manufacture of the blocks. Subsequently, the base material is conveyed by conveyor belts to the vibratory press so that the necessary shaping and final production of the blocks occur.

Finally, the brushing and elimination of the chips are performed, analyzing if they are following the norms. The structural part of the block (compressive strength and water absorption) meets the specifications of ABNT NBR 6136: 2010, NBR 12118: 2012, and NBR 9781: 2013. Given this, the criteria can be defined to achieve maximum productivity in the manufacture of concrete blocks from the foundry sand, helping managers decide.

3.3. Definition and Criteria

Given the analysis and observation, together with the managers of the production process, four criteria were defined that will be used to improve the efficiency and productive capacity of the organization. As shown in Figure 1, the behavior of the inlets in relation to the outlet was observed, that is, the influence of each one to optimize the productivity in the production of concrete blocks from the foundry sand.

3.4. Fuzzy Simulations

With the help of managers, simulations were performed, from which the limitations for analyzing the criteria in relation to quality in the production process were elaborated. Thus, the rule base for a collection of fuzzy propositions. Therefore, all possible combinations were used to give more accuracy to the application of the method.

The temporality dimension is of paramount importance as it relates between the time of entry of the record in the databases and the time of occurrence of the event where a service is completed. Moreover, the time between the occurrence of production events and the

availability of relative information to users can also be considered.

Therefore, once the constraints were determined, simulations were obtained, as shown in Figure 2, to observe the production behavior, considering the cost and time variables. Detailed cost analysis, production cost calculation, loss quantification efficiency estimation provides a solid basis for financial control. Managers must also be concerned about future costs, their level of supply and production decisions, and pricing policies.

It is noticeable that the behavior of the variables is not constant, showing that there are changes when the time in days is increased about the cost, thus helping the manager to note the best time with the lowest cost. Figure 4 shows the cost and equipment variables concerning production. It can be seen that the manager can understand the correct number of machines to use to reduce their costs.

Figure 4 shows the constant development of companies and their production processes seeking to reduce costs through the amount of human resources needed to produce effectively and efficiently. Figure 4 shows the behavior of production to the variables cost and human resources. Controlling variables such as human resources and equipment efficiently and effectively leads to better results at lower costs. Figure 5 shows the relationship of production to human resources and equipment.

Regarding the variable human resources related to equipment, the manager analyzing the figure may decide how staff to use not to have staff stopped generating less productivity. Figure 6 shows the behavior of production with human resources and time. Thus, the figure above shows the time to human resources, thus denoting the best values to improve production, helping the manager verify the time to be spent and the number of employees needed to improve productivity. In Figure 7, one can see the behavior obtained in production when compared to time and equipment.

Thus, improvements and automation in the data collection process can also contribute to a good performance of these measures; therefore, these results help managers in decision making to improve the production of concrete blocks from the ADF. According to the simulations performed, it was evidenced that, to reach an optimization in production, one must work with a cost of R\$ 67.700.00, optimizing its production time to approximately 18 days, where previously it was 30

days, still using about 65% of its equipment and about 2 of its employees. Doing a new survey in the company found that the first work has been implemented and aims to reach the maximum capacity. The managers showed that from 2017 until June 2018 began to use 90% of production capacity, and previous data showed 80% of it.

4. CONCLUSIONS:

The objective of the present work is to optimize the productive capacity of concrete blocks from foundry sand using fuzzy logic. Thus, finding managers a possible alternative in decision making for the future of the enterprise. The same has been achieved given that the organization has great market potential, as it is a pioneer in its region to use foundry sand in the manufacture of blocks. It emphasizes a vision of sustainability established by the company by reusing harmful materials and presenting a possibility for the disposal of industrial waste generated by the mechanical manufacturing industry.

However, it is worth noting that the study is a simulated proposal for multiple reasons, both internal and external, which may lead to changes in the results. Moreover, it is noteworthy that the fuzzy sets methodology strongly supports decision-making, which comes against the solution of subjective and intuitive criteria widely used by experts (Hisrich and Jankowicz, 1990; Mitchell *et al.*, 2005).

Thus, the company understudy has a lot to gain, because the evidence helps in the decision making increasingly safe, therefore using the resources effectively, thus avoiding waste and idle time, which is one of the biggest problems diagnosed in the company. However, the company is open to suggestions denoting; therefore, future plans intend to reach the maximum plant load that is 100%, target for the next six months. The company seeks to develop reaching the goals of improvement and efficiency and thereby gain an effective production process.

As a limitation found in the research, it is controversial that managers spontaneously corroborate the adopted strategies regarding the reliability of data collection. An imperfect way to overcome such an obstacle would be the empirical separation of the intensity of adoption of the relevant variables, based on publicly available related information. Another limitation worth mentioning is the time to conduct the interviews. The managers often did not have the time because they were busy doing their work in the company's

administration and could not receive the researchers.

Based on the above, as a suggestion for future work, it is indicated a study on the effects of casting sand compared to river sand on the mechanical properties and on the microstructure of these concretes. It is also indicated, a study comparing the fuzzy topsis methods and the ANP, to analyze the sectors of the company regarding the productive system, defining a hierarchy of action, thus solving the possible existing bottlenecks.

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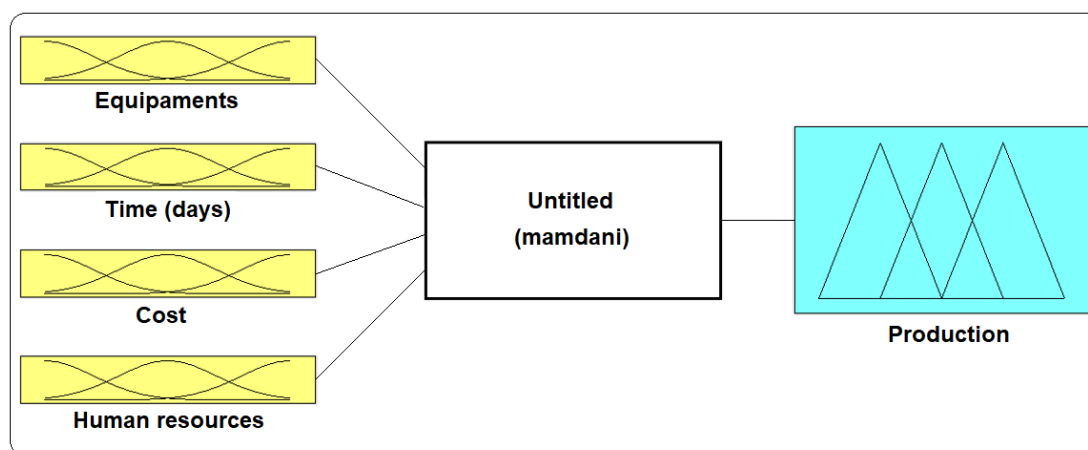


Figure 1. Simulation inputs and outputs for production quality improvement

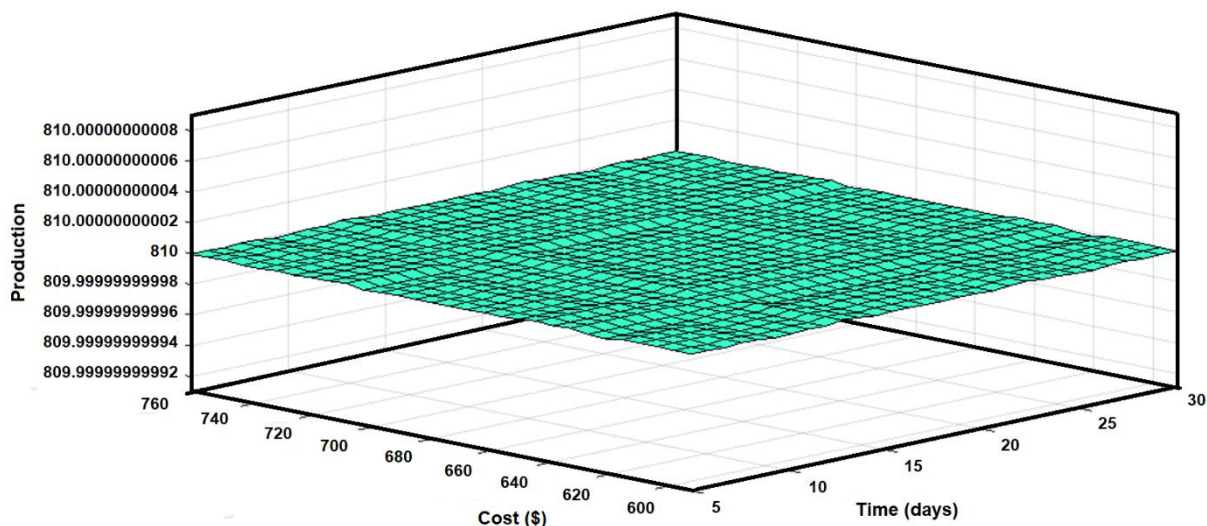


Figure 2. Cost vs. Time

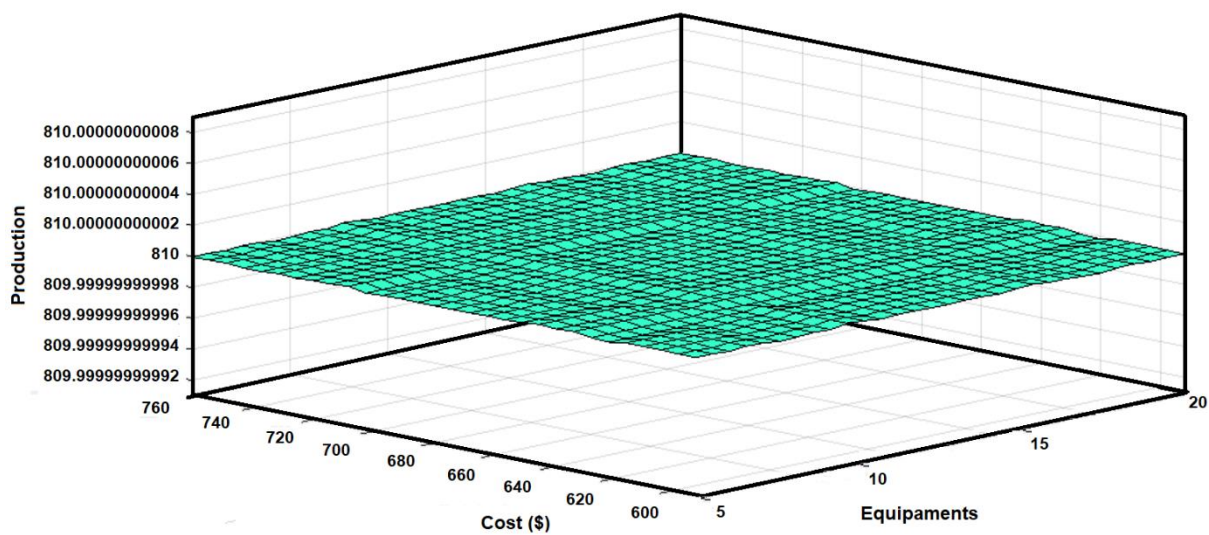


Figure 3. Cost vs. Equipment

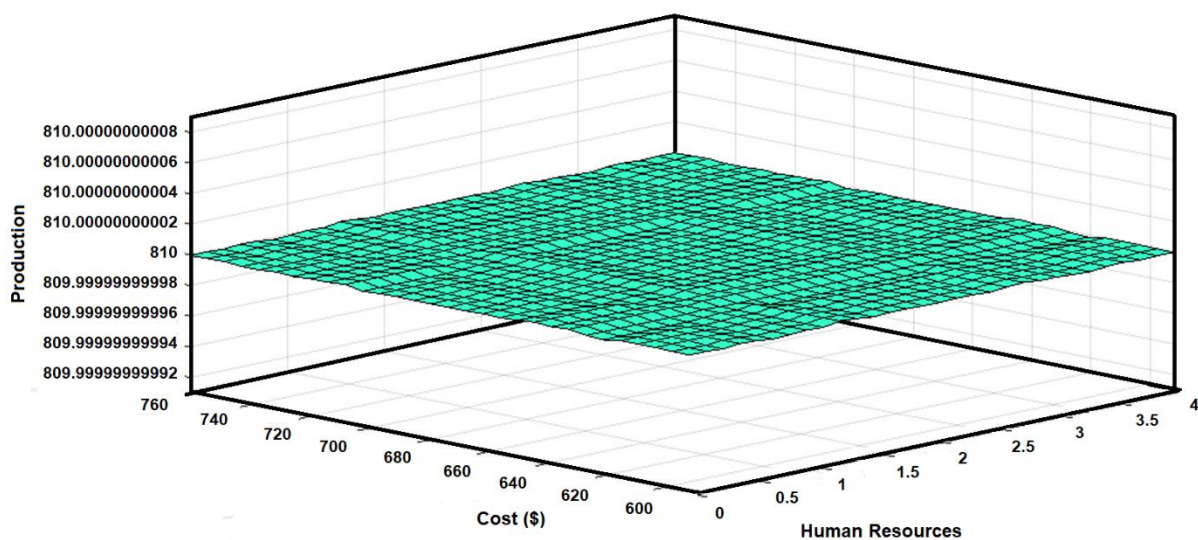


Figure 4. Cost vs Human Resources

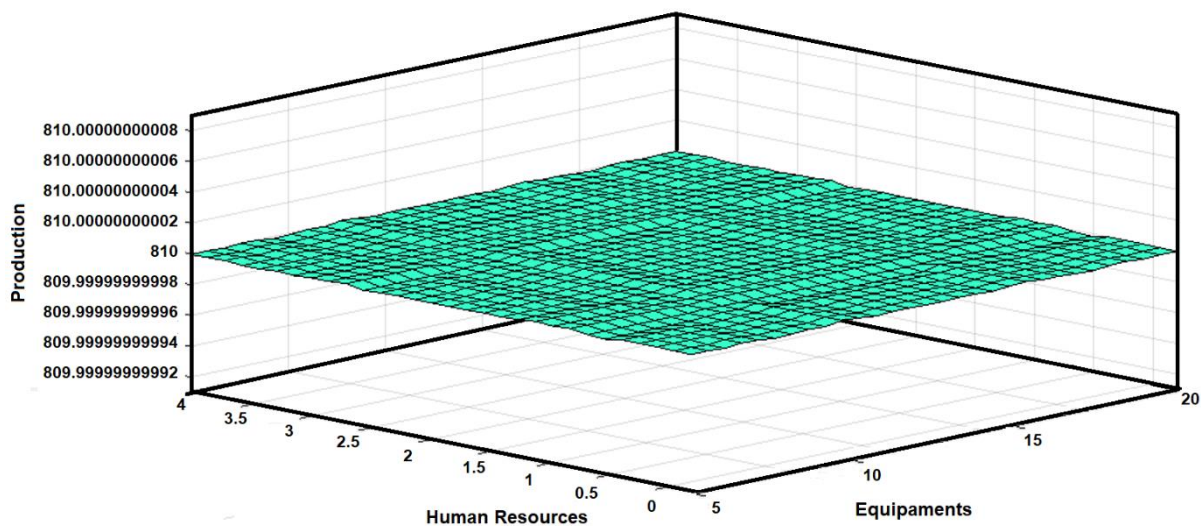


Figure 5. Human Resources vs. Equipment

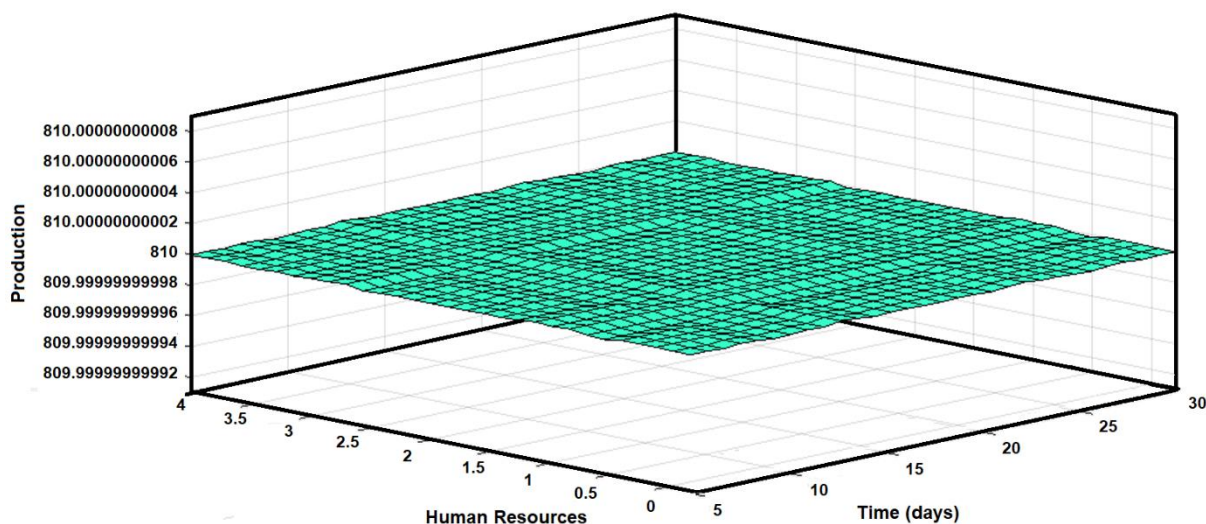


Figure 6. Human Resources vs. Time

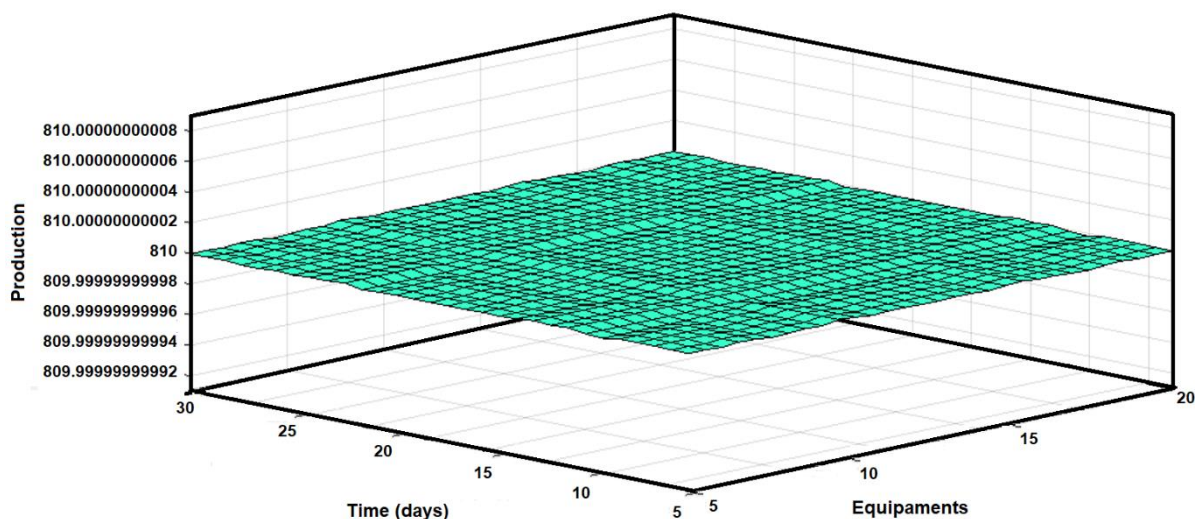


Figure 7. Time vs Equipment

E-SELECTIN AS A BIOMARKER IN FEMALE PATIENTS WITH B-THALASSEMIA IN AL- NAJAF PROVINCE, IRAQ

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ABSTRACT

E-selectin, as identified (CD62E), is expressed on endothelial cells after stimulation with inflammation cytokines. β -Thalassemia diseases (β T) and early diagnosis are of utmost significance in the entire world population. This study was performed in the Thalassemia Center of the Al-Zahraa Educational Hospital in Al-Najaf Province, Iraq, on sixty-nine with β -thalassemia (54 β T major and 15 β T Intermedia) aged 8-40 years who transfused blood. Compared to 20 healthy volunteers as a control group. In both β T patients and healthy groups were assessed serum E-selectin levels. It was investigated the relationship with RBC, Hb, PCV, WBC, PLT, BMI, splenic status, iron, and ferritin levels. The results revealed a significant ($P<0.05$) decreased values of HB, RBC, P.C.V, and BMI. In contrast, values of WBC, PLT, Iron, and Ferritin were significantly increased in β T patients as compared to the healthy control groups. A significant ($P<0.05$) increase in serum E- Selectin level in β T patients (20.55 ± 0.47) ng/ml to compare with the healthy group (9.16 ± 0.50) ng/ml. Furthermore, it was a significant decrease in groups of β T major (19.87 ± 0.42) ng/ml more than in β T intermedia (23 ± 1.42) ng/ml. E-Selectin revealed a significant increase ($P<0.05$) in progress age and associated with splenectomies and underweight groups compared to splenectomies and the normal weight groups, respectively. Also, E-Selectin levels significantly positively correlated with WBC, PLT value, iron, and Ferritin levels. However, it was no significant with RBC, PCV, Hb. As a conclusion from this study, E- Selectin is an important biomarker in β -thalassemia patients can be identified as the complications associated with iron overload, inflammatory process, and endothelial dysfunction in β T disease.

Keywords: *E- Selectin, Inflammation cytokines, Iron overload, and endothelial dysfunction*

1. INTRODUCTION

Thalassemia is one of the world's main hemoglobinopathies in the population. In human β -thalassemia (β T), a single gene inherited hemoglobin disease, an autosomal recessive disorder caused by diminished or absent development of β globin chains. (Lettre, 2012; Bernard *et al.*, 2013).

Anemia in thalassemic patients has a significant effect on increasing damage to the cytoskeleton proteins of the RBC membranes, leading to intravascular hemolysis, particularly in the spleen, and thus to the regular intramedullary destruction of red cells. (Thein, 2013).

A previous study has shown that hypoxemia, activation of leukocytes, and platelets may partly contribute to anemia that may influence

endothelial function and raise levels of cell and endothelial adhesion markers. (Ibrahim *et al.*, 2013b).

E-selectin (CD62E) is a cell adhesion molecule for membrane glycoproteins. It is one of three selectin-endothelial forms (E-, P-, and L-selectins) (McEver and Zhu, 2010). After induction by inflammatory cytokines, it is expressed on endothelial cells, and the slow rolling of leukocyte adhesion to the vascular endothelium mediates efficiency. E-selectin represented endothelial dysfunction resulting from oxidative stress in many inflammatory diseases (Chase *et al.*, 2012; Ibrahim *et al.*, 2013a).

This study aimed to investigate serum E-selectin levels and their relationship with hematological and biochemical parameters associated with iron overload in β -thalassemia diseases.

2. MATERIALS AND METHODS

2.1. Study design

This study was supported by the approval of the ethical committee regulations of Kufa University. The study included sixty-nine females with β -thalassemia (54) β -thalassemia major and (15) intermedia β - β -thalassemia, the aged range from (8–40) year. They were visited at The Thalassemia Center in Al-Zahraa Educational Hospital in Al-Najaf province, Iraq. They were presently transfused and treated every 2-3 weeks for the clinical signs and manifestations of the disease that involved blood transfusion, which was about (10 ml of blood for each kilogram of body weight, to keep of Hb level leastwise 10 g/dl), and regulation iron-chelating therapy, where were considered as a target for the present study. Also, (20) healthy females who did not suffer from any disease served as a control group; the patient and controls were age-matched. This study was carried out from July 2014 to March 2015. Divided into β -thalassemia patients according to

2.1.1. Types of β -thalassemia divided into two groups

β -Thalassemia Major n=54, and β -Thalassemia Intermedia n =15

2.1.2. The aged of β -thalassemia divided into three groups:

Group 1 [(8 –18 year), n =51], Group 2 [(19 –29 year), n =12], and Group 3 [(30 – 40 year), n =6].

2.1.3. Splenic situation divided into two groups:

Patients with Splenectomized n=29 and Unsplenectomized patients n=40.

2.1.4. Body Mass Index (BMI) divided into two groups:

Patients with normal weight n=46 and Underweight patients n=29. The normal range (5th – 85th) % for (≤ 20) years of age, and (18.5 – 25) kg/m² for (>20) years of age. (WHO, 2006)

2.2. Methods

2.2.1. BMI (Body Mass Index)

An electronic balance and height unit carried out the BMI measurement to measure weight divided on the height square and Equation 1 was applied (WHO, 2006).

$$\text{BMI} = \text{Weight (kg)} / \text{Height (m}^2\text{)} \quad (\text{Eq. 1})$$

In children, BMI is compared against the percentile for children of the same gender and age, the standard values in adults and children (CDC, 2014).

2.2.2. Hematological Assessments

At Hematology Laboratory of Al-Zahraa Educational Hospital, both patients and healthy were withdraw 5 ml of venue blood. Hematological criteria were conducted on EDTA anticoagulated blood by using a completely automated hematology analyzer Mythic 18 (RINGELSAN CO., Turkey) for estimated complete blood count (CBC) (Wasmuth, 2010).

2.2.3. Estimation of Serum Iron concentration

Serum iron concentration was measured by iron (using the chromogen ferrozine method) kit (bt 35i, Turkey). The principle transferrin-bound iron is released at acid pH and reduced from ferric Fe³⁺ to ferrous ions Fe²⁺ iron. These ions react with ferrozine to form a violet-colored complex, which is measured spectrophotometrically at 560 nm. This absorbance is proportional to serum iron concentration in the sample, and Equation 2 was applied (Persijn *et al.*, 1971).

$$\text{Total iron } (\mu\text{g/dl}) = [(\text{Abs. 2 Sample} - \text{Abs.1Sample}) / (\text{Abs. 2 Std.} - \text{Abs. 1 Std.})] \times \text{Conc. Std.} \quad (\text{Eq. 2})$$

Abs. = Absorbance, Std. = Standard

2.2.4. Estimation of Serum Ferritin

This methodology was created to measure ferritin serum levels using the enzyme-linked immunosorbent assay (ELISA) immunoenzymatic technique using the bioelisa ELx 80000 reader (biokit, U.S.A). According to the Manufacturing Firm, the Human Accu Bind Ferritin ELISA Kit (Monobind Inc., U.S.A, code number 2825-300). (Anderson and Kelly,1981).

2.2.5. Estimation of serum E-selectin level

Evaluating of Human E-Selectin ELISA Kit was executed as mentioned by the manufacturing company (CUSABIO BIOTECH Co., Ltd., P.R.C, code number CSB-E04540h) that depended on the technique of the quantitative sandwich enzyme immunoassay (Koch *et al.*, 1995).

2.4. Statistical analysis

The data were evaluated statistically

through the SPSS bundle (SPSS, Version 17). Descriptive analyses between the patients and control groups, also among patients. Data represented at mean \pm standard error of the mean. The distinction between subdivided classes in the calculated parameters, Pearson correlation, and multivariate ANOVA was used, while the figures constructed using the Microsoft Office 2013 EXCELL software were used. All of these were evaluated statistically at a significant $p < 0.05$ level.

3. RESULTS AND DISCUSSION:

3.1. General criteria of the study subjects

The general characteristics of the studied groups were shown in Table 1. Each category of β -thalassemia patients can be identified approximately by hematological and biochemical parameters. The results revealed a significant difference ($p < 0.05$), decreased levels of HB, RBC, and P.C.V, were found in β -thalassemia patients (7.7 ± 0.14) g/dl, (3.13 ± 0.07) 10^6 /ml, and (23 ± 0.41) % as compared to the healthy control groups (12.01 ± 0.19) g/dl, (4.36 ± 0.07) 10^6 /ml, and (36.34 ± 0.52)% respectively. WBC, PLT count values in the patients of β -thalassemia (11.05 ± 0.26) 10^3 /ml, (361.86 ± 13.92) 10^3 /ml exhibited highly significant ($p < 0.05$) as compared to the healthy group (7.59 ± 0.33) 10^3 /ml, (244 ± 14.23) 10^3 /ml. The results revealed a significant decrease ($p < 0.05$) in BMI of β -thalassemia patients (16.59 ± 0.43) kg/m² comparison to (22.15 ± 0.36) kg/m² in the healthy control groups.

Iron and ferritin were statistically significant increase noted in β -thalassemia patients (172.2 ± 4.4) μ g/ml, and (5156.5 ± 438.7) ng/ml as compared to the healthy control groups (29.86 ± 2.32) μ g/ml, and (99.15 ± 8.95) ng /ml respectively. The hematological parameter (Table 1) in the present study showed a substantial decrease in Hb, PCV, and RBC levels, in addition to a significant increase in WBC and PLT in the number of female patients with β -thalassemia. Current findings agree with Arshad *et al.* (2014), who reported that, due to the reduction of erythrocyte numbers and reduced RBC index values, Thalassemia patients might have anomalies associated with lower Hb level (MCV, MCH, MCHC, HCT). Thus, these patients suffer from anemia, which results in reduced blood oxygen content. Some research has shown that reduced amounts of Hb, PCV, and RBC counts detected in patients with β -thalassemia are attributed to their early deterioration and persistent

erythrocyte dissolution due to an irregular globin molecule contributing to erythrocyte breakup before maturation (Shanthi *et al.*, 2013). In patients with β -thalassemia, a reduced erythrocyte lifetime and premature cell death are essential theories of accrual of free extra globin chains within the RBC membrane surface, inducing oxidative stress to create a free radical that causes damage to the RBC membrane (Ibrahim *et al.*, 2013b). The findings of this analysis are acceptable, with recent research referring to results related to RBC mass, WBC, and a significant difference in PLT count in patients with β -thalassemia. This is due to continued extreme anemia followed by hypercellular (thrombocytosis and leukocytosis) triggered by activation of the erythropoietin hormone that works on the bone marrow to enhance the distribution of blood cells or to stimulate the immune system by receiving blood from different donors (Yassin *et al.*, 2013; Arshad *et al.*, 2014). Present findings of this study showed that patients with β -thalassemia had a substantial drop in BMI, and almost two-thirds (66.7 %) were underweight and (33.3 %) average weight relative to stable control (Table 1). These findings corresponded to those of (Eissa and El-Gamal, 2014). They reported that BMI and iron overload resulted in ferritin being proposed as low BMI markers for patients with β -thalassemia. Many studies have suggested that the pathogenesis of β -thalassemia growth deficiency is multifactorial such as systemic diseases, endocrinopathies secondary to iron overload, and Deficiency in essential dietary elements resulted from iron overload, which Important contributing factors to the development of underweight patients (Skordis, 2011; Manali *et al.*, 2015).

3.2. Serum E-selectin level and correlated with the hematological and biochemical parameters

The results were showed in (Figure 1) indicated that a significant ($p < 0.05$) increase in serum level of E-Selectin patients with β -thalassemia (20.55 ± 0.47) ng/ml, in comparison with that of control groups (9.16 ± 0.50) ng/ml. These were accepted with (Kanawaki *et al.*, 2012), While it was a significant decrease in significant β -thalassemia (19.87 ± 0.42) ng/ml, to compare with that of intermedia β -thalassemia (23.00 ± 1.42) ng/ml, (Figure 2). Therefore, Serum E-selectin showed a significant decrease ($P < 0.05$) in the age group (8-18) about (19.16 ± 0.40) ng/ml in comparison with the age group (19-29) and (30-40) about (22.11 ± 0.59) ng/ml and (29.2 ± 0.58) ng/ml, respectively. Moreover, it presented the same significant decrease when it compared of the age group (19-29) about (22.1 ± 0.59) ng/ml, less

than the age group (30-40) about (29.2 ± 0.58) ng/ml, (Figure 3). The results indicate a significant increase ($p < 0.05$) in E-Selectin level in β -thalassemia patients with splenectomized (22.13 ± 0.63) ng/ml, in comparison with unsplenectomized (19.4 ± 0.61) ng/ml (Figure 4). Moreover, it was a significant decrease ($p < 0.05$) in patients with underweight (19.30 ± 0.42) ng/ml to compare with normal-weight patients (23.04 ± 0.95) ng/ml (Figure 5).

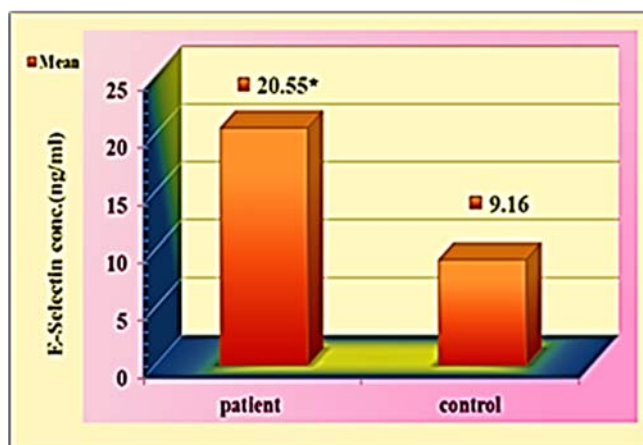


Figure 1. serum E- selectin level in β T patients compare with the healthy groups as control. * statistically significant at $p < 0.05$

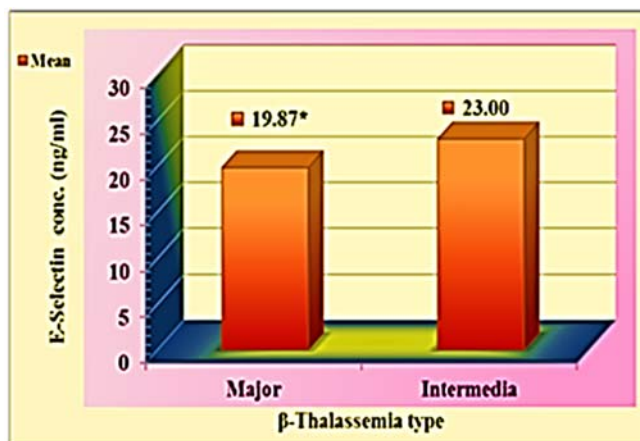


Figure 2. Serum E- selectin level in β T major compare with β T- intermedia groups. * statistically significant at $p < 0.05$

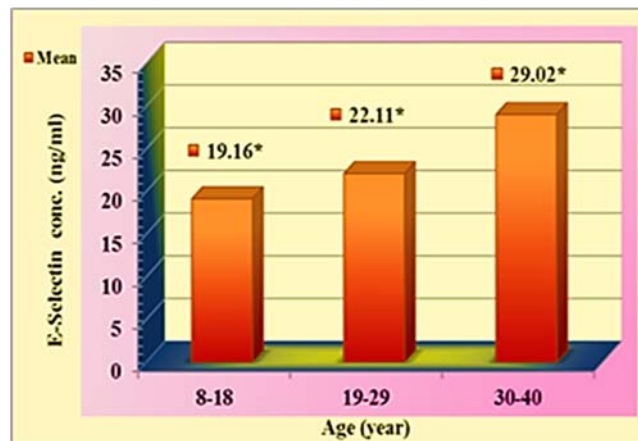


Figure 3. serum E- selectin level in different age groups of β T patients. * statistically significant at $p < 0.05$

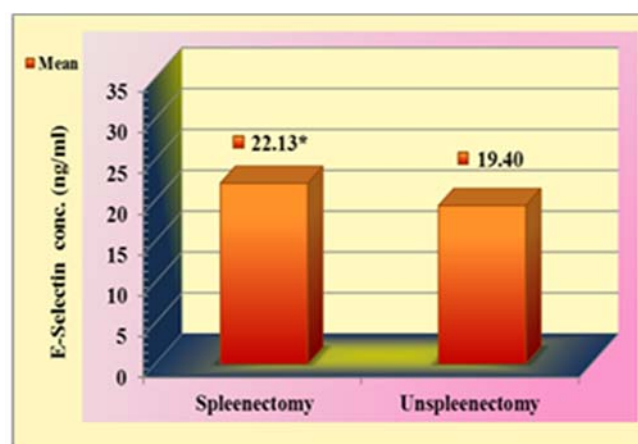


Figure 4. Serum E- selectin level in β T patients with splenectomy and unsplenectomy groups. * statistically significant at $p < 0.05$

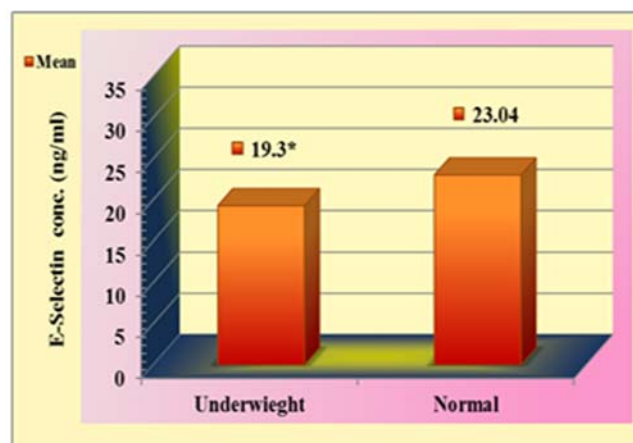


Figure 5. Serum E- selectin level in β T patients with underweight and normal-weight groups. * statistically significant at $p < 0.05$

The results of correlated showed that serum E- selectin level no significant correlated with RBC ($R^2 = 0.007$), PCV ($R^2 = 0.006$), and HB ($R^2 = 0.002$), (Figure 6 a, b, and c). respectively.

In addition, it was a positively significantly correlated with WBC ($R^2 = 0.203$) and, PLT ($R^2 = 0.117$), Iron ($R^2 = 0.091$) and Ferritin ($R^2 = 0.168$), (Figure 6 d, e, f, and g), respectively.

E selectin is exclusively expressed on the surface of endothelial cells in a highly inductive manner in response to inflammatory cytokines, endothelial E-selectin assists in the initial adhesion of circulatory leukocytes to the surface of vascular endothelium. Some studies were showed that chronic inflammatory is more prevalent in thalassemia patients; because the oxidative stress resulting from iron overload plays an essential role in endothelial vascular activation of adhesion molecules and induction of monocyte and neutrophil in the inflamed site (Taher *et al.*, 2011; Vinchi *et al.*, 2013),

Endothelial dysfunction is due to intravascular hemolysis, leading to proliferative vasculopathy. Increased adhesion molecules such as E-selectin on the vascular endothelium, high levels of inflammatory cytokines and stimulated leukocytes, significant amounts of ROS, and promote thrombus (Stoyanova *et al.*, 2012). The present study (Figures 2 and 4) showed a significant increase of serum E-selectin level in intermedia and splenectomized groups compared with thalassemia major and non-splenectomized groups, respectively. These were in agreement with (Atichartakarn *et al.*, 2014). Who found that splenectomized patients had a more severe hemolytic disease and higher levels of E- P-selectins. which suggested that intravascular hemolysis (IVH) and vascular endothelial cell (EC) activation were occurred by chronic iron overload and activation of the chronic inflammation (Taher *et al.*, 2011) has been explained that among the medical complications of thalassemia intermedia that found to occur at higher rates. Even more frequently than in patients with thalassemia major. Splenectomized and transfusion were significant risk factors for thromboembolism events, which increased in patients with thalassemia intermedia. An increase of serum E-selectin level with increasing age, resulting from repeated blood transfusion increase with age progress, leads to increases free radical production and oxidative stress of tissues, leading to elevated release endothelial molecules which included E-selectin. This finding agrees with the study of (Sena *et al.*, 2013), showing that aging is associated with increased oxidative stress and a proinflammatory endothelial cell phenotype, and excessive or prolonged endothelium activation due to the action of proinflammatory cytokines underlies

endothelium dysfunction (Zinovkin *et al.*, 2014) Study of Hisham *et al.*, (2013) illustrated that high levels of E-selectin in the serum following a high level of leptin, and other study showed that serum levels of leptin in major beta-thalassemia reduce regardless of age and body mass. Also, there was a positive relationship between serum leptin level and BMI in patients with significant beta-thalassemia (Shahramian *et al.*, 2013). Previous studies have shown that serum E-selectin levels are a high expression in obesity and decline with reducing weight. They found an increased layer of visceral adiposity maybe a companion with E-selectin, then related to BMI (Pontiroli *et al.*, 2009; Zanni *et al.*, 2011).

4. CONCLUSIONS:

E-selectin can be released into the circulation and quantified in plasma and serum, reflecting by these inflammatory processes within vascular cell walls and marker of endothelial dysfunction, also plays an important role in the recruitment of WBC, RBCs, PLT, and promote thrombosis at vascular inflammation sites (McEver and Zhu, 2010; Taher *et al.*, 2011; Stoyanova *et al.*, 2012). Previously study illustrated that heme from RBC lyses circulated in the blood and led to the expression of endothelial adhesion molecules, causing increased adhesion of leukocytes and reticulocytes endothelium in hemoglobinopathy (Wagener *et al.*, 2001).

The current study concluded that elevated serum E-selectin level in β -thalassemia patients, especially in patients with splenectomized and underweight, and development with progressed age associated with the inflammatory process and iron overload. This study suggested serum E-selectin as a potential biomarker for the early diagnosis of important complications related to endothelial dysfunction.

5. ACKNOWLEDGMENTS:

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Table 1. The clinical characteristics in female patients with β -thalassemia Compare to the healthy groups

Clinical characteristics	β T Patients n= 69	Control n= 20	P-Value
Age (year)	16.62 \pm 0.96	20.75 \pm 1.86	0.612
RBCs (10 ⁶ /mm ³)	3.13 \pm 0.07 *	4.36 \pm 0.07	0.038
Hb (g/dl)	7.7 \pm 0.14 *	12.01 \pm 0.19	0.027
P.C.V %	23 \pm 0.41 *	36.34 \pm 0.52	0.036
WBCs (10 ³ /mm ³)	11.05 \pm 0.26 *	7.59 \pm 0.33	0.036
PLTs (10 ³ / mm ³)	361.86 \pm 13.92 *	244 \pm 14.23	0.034
BMI (kg/m ²)	16.59 \pm 0.43 *	22.15 \pm 0.36	0.001
Underweight	13.09 \pm 0.52	0	
Normal weight	23.7 \pm 2.04	0	
IRON (μ g/ml)	172.2 \pm 4.4 *	29.86 \pm 2.32	0.0001
FERRITIN (ng /ml)	5156.5 \pm 438.7 *	99.15 \pm 8.95	0.0001

* P< 0.05 statistically significant with the control group. Red Blood Corpuscular - RBCs. Hemoglobin – Hb. Packed Corpuscular Volume - P.C.V. White Blood Cells – WBCs. Body Mass Index – BMI. Platelets – PLTs. Data represented as mean \pm standard error of mean.

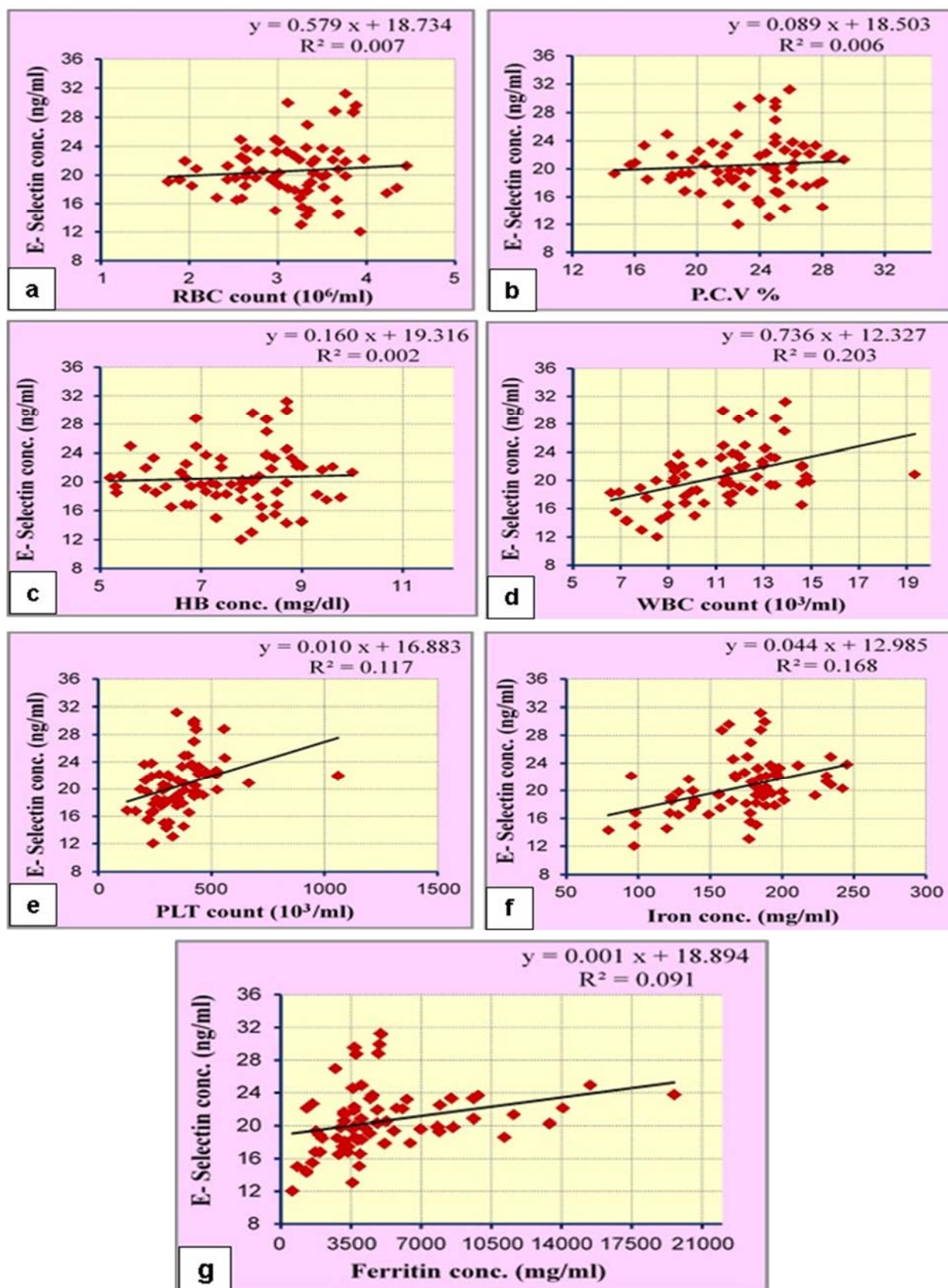


Figure 6. Scatter pots of serum E- selectin level correlated. No significant with RBCs (a), P.C.V. (b), and Hb (c). A positive significance with values of WBCs (d), PLTs (e), iron (f), ferritin (g)

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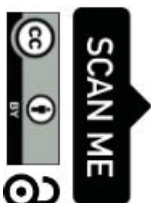
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Conclusion: Summarize the data discussed in the Results and Discussion showing the relevance of the work and how different it is from other researches. Also, point out the benefits and improvements that can be observed in order to develop new science standards that can change something in the related field.

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2. Provstyanov, M. V.; Logachev, E. V.; Kochergin, P. M.; Beilis, Y. I.; Izv. Vyssh. Uchebn. Zaved.; Khim. Khim. Tekhnol. 1976, 19, 708. (CA 85:78051s).

If the article has a DOI number, but its complete reference is not given, the DOI number should be cited as follows:

3. Vidotti, M.; Silva, M. R.; Salvador, R. P.; de Torresi, S. I. C.; Dall'Antonia, L. H.; Electrochimica Acta (2007), doi:10.1016/j.electacta.2007.11.029.

It is recommended to give composite references instead of a list of separate references. The style of composite references is as follows:

4. Varela, H.; Torresi, R. M.; J. Electrochem. Soc. 2000, 147, 665; Lemos, T. L. G.; Andrade, C. H. S.; Guimarães, A. M.; Wolter-Filho, W.; Braz-Filho, R.; J. Braz. Chem. Soc., 1996, 7, 123; Ângelo, A. C. D.; de Souza, A.; Morgon, N. H.; Sambrano, J. R.; Quim. Nova 2001, 24, 473.

Patents

Patents should be identified as follows (if possible, the Chemical Abstracts number should be given in parentheses):

5. Hashiba, I.; Ando, Y.; Kawakami, I.; Sakota, R.; Nagano, K.; Mori, T.; Jpn. Kokai Tokkyo Koho 79 73,771 1979. (CA 91:P193174v)
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With editors

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8. Regitz, M. Multiple Bonds and Low Coordination in Phosphorus Chemistry; Regitz, M.; Scherer, O. J., eds.; Georg Thieme Verlag: Stuttgart, 1990, cap. 2.

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9. Cotton, F.A.; Wilkinson, G.; Advanced Inorganic Chemistry, 5th ed., Wiley: New York, 1988.

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10. Sheldrick, G. M.; SHELXL-93; Program for Crystal Structure Refinement; University of Gottingen, Germany, 1993.

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13. <http://www.sbj.org.br/jbcs>, accessed June 2001. (minimal of information requested)

Unpublished material

For articles accepted for publication: Magalhães, U. H.; J. Braz. Chem. Soc., in press.

For articles submitted but not yet accepted: Magalhães, U. H.; J. Braz. Chem. Soc., submitted.

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НАУЧНАЯ КОНФЕРЕНЦИЯ

ДЕКАБРЬ 2021

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DEZEMBRO 2021

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CONFERENCIA CIENTÍFICA

DICIEMBRE DE 2021

La CONFERENCIA VIRTUAL 2021 DE LA REVISTA DE QUÍMICA DEL SUR DE BRASIL es un evento cultural organizado por la Revista de Química del Sur de Brasil, el Periódico Tchê Química y otros socios, para celebrar los casi 30 años de la Revista de Química del Sur de Brasil.

El objetivo de la conferencia es proporcionar a los autores un lugar para presentar sus comunicaciones científicas en múltiples formas, como la presentación de pósters, la publicación de un manuscrito completo, la publicación de un resumen ampliado y/o presentación de un póster pregrabado (hasta 5 minutos de duración).



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