`Vol. 5, No. 1, pp. 101-115, (March 2024)
DOI: 10.21608/aujes.2024.259964.1207
Aswan University Journal of Environmental Studies (AUJES)
Online ISSN: 2735-4237, Print ISSN: 2735-4229
Journal homepage: https://aujes.journals.ekb.eg/ E-mail: AUJES@aswu.edu.eg
Original research

Descaling treatment of sugarcane Evaporative tubes using Organic acids solutions

Safaa El-Nahas*[1], Mahmoud Kodari[1], Ali A. Hamam[1,2] and Ahmed N. Gad El Rab[3]

[1]*Chemistry Department, Faculty of Science, South Valley University, Qena 83523, Egypt.*
[2] *Arment sugar factory, Luxor, Egypt.*
[3]*Research & development unit, Quos sugar Factory, Qena, Egypt.*

Received: 1/1/2024 Accepted: 21/1/2024

Abstract:

Scales are mechanically removed from sugar evaporators' pipes and equipment. Although effective, this method is usually costly, difficult to access deposits in crevices, and risks causing equipment damage. Chemical cleaning, on the other hand, is a more effective, trustworthy, and time-efficient alternative for eliminating scale. A solution of caustic soda (10-25%) is the most used reagent in Egypt to clean the evaporative tubes used in sugar processes. The goal of this study was to provide a novel green solution that could be utilized in sugar factories to remove scale from evaporative bodies and reduce the environmental impact of NaOH. Consequently, glutamic acid and sodium gluconate were both explored as organic acids for scales dissolution. In all types of evaporator bodies, glutamic acid has a significantly better removal efficacy of up to 83%, while sodium gluconate gave a removal rate of up to 40%. According to EDX and XRD analysis, the main scales content in the sugar factory was calcium sulphate due to the sulphitation method utilized in the clarification operations. The fourth evaporator body is the most difficult because to the high quantity of Si contained in the scale structure. The findings of this study will help the industry sector choose precise thinning techniques for the use of chemical chelating agents for scale removal.

Keywords: Scale –cleaning solution – sugar Factory

1- Introduction

Scale issues cost the business hundreds of millions of dollars in production costs and losses each year. Equipment life can be significantly extended by increasing the frequency of chemical cleaning or limiting the scale building process (Gleick, 2003). More than 80% of industrial heat exchangers, which are used in the pulp and paper, oil, gas, chemical, and petrochemical industries as well as the food, beverage, and water purification industries, have fouling problems. Scaling generates a variety of troubles including a rise in energy consumption, equipment shutting down or malfunctioning, and heat transfer impedance (Popuri et al., 2014).

Corresponding author*: E-mail address: Safaa.elnahas@sci.svu.edu.eg/

Along with limiting heat transfer, fouling in heat exchangers can also reduce mass transmission. Fouling is usually understood to be the buildup and development of undesirable materials on the surfaces of processing equipment. The phenomenon of fouling is incredibly complex (Awad, 2011). Since the ninth and tenth centuries, Egyptians have been considered pioneers in the refined sugar industry. In 2007-2008, Egypt produced 1,582 million tons of sugar while using 2,485 million tonnes. Sugarcane, on the other hand, is planted in 136 million hectares and yields 1075184 metric tons of sugar. This assessment discusses the latest progress and distribution of Egypt's sugar beetroot and sugarcane industries (Hassan and Nasr, 2008). One of the sugar manufacturing processes is the clarification which is chemical treatment of the cane sugar juice to purify juice form both the colloidal fine, suspended matter and impurities. Purification is required so that pure sugar crystals can be obtained later. Also, purification of the juice prevents decomposition of sucrose by the action of impurities. Thus, fine suspensions are coagulated giving particles which will settle at the reasonable rate.

Sulphitation process at sugarcane factories consists of the addition of (SO_2) gaseous into the juice until pH 3.8 – 4.2 is reached subsequently, neutralization (pH 7.0 – 7.2) by addition $Ca(OH)_2$ hydrated lime. The alkalization of the sulphate juice led to the formation of calcium sulphate, which adsorbs undesirable compounds from the juice (Hugot, 2014) **.** The combined effect of the lime and heating is to form flocculent precipitate which entrains much of the very fine suspended matter and on settling leaves a clear juice. Besides, the action of sulphur dioxide on juice is bleaching neutralizing and viscosity reducing (Honig, 1953)**.** In the process of removing water from a solution by vaporization through the application of heat is needed. In other words, we can say that evaporation is a type of vaporization of a liquid that occurs from the surface of a liquid into a gaseous phase that is not saturated with the evaporating substance (Kulkarni, et al., 1996). The term "single effect evaporator system" refers to the use of a single evaporator to concentrate any solution, and the term "multiple effect evaporator system", (Figure 1) refers to the use of numerous evaporators in series to concentrate any solution (Kahsay and Gabbiye, 2015). The production of scales fouling in sugarcane and sugar beet is a major worldwide agricultural industry**.** Pre-heaters, distilleries, and evaporators almost always scale, but to varying degrees at different sugar facilities. These encrustations are the result of several chemical reactions involving ions or molecules that are both organic and inorganic. Scaling may be brought on by contaminants in sugar juice or processing water (Firdissa, 2012). Due to its poor thermal conductivity, scale's accumulation on metal surfaces prevents heat from being transferred through a tube's wall, encourages the corrosion of surfaces, and inhibits fluid movement.

The type of scale that is generated is influenced by a variety of factors, including the quality of the raw materials and intermediate products, the fluid's flow characteristics, the rate of evaporation, and the system's operating circumstances (Cao, 2010). Caustic soda is the most commonly used chemical for cleaning, or a mixture of caustic soda and soda ash. In some cases, particularly the early effects, it can be used on its own. A concentration of >20% solution is usually required, while in some other cases caustic soda concentrations may be ranged between 10 and 40 g/100g as reported (Walthew, et al., 1997). High caustic concentrations also make boiling more difficult because of the high boiling point elevation. Consumption of caustic is in the range of 10 to 30 kg/100 m^2 surface area (Rathoure, et al., 2019).

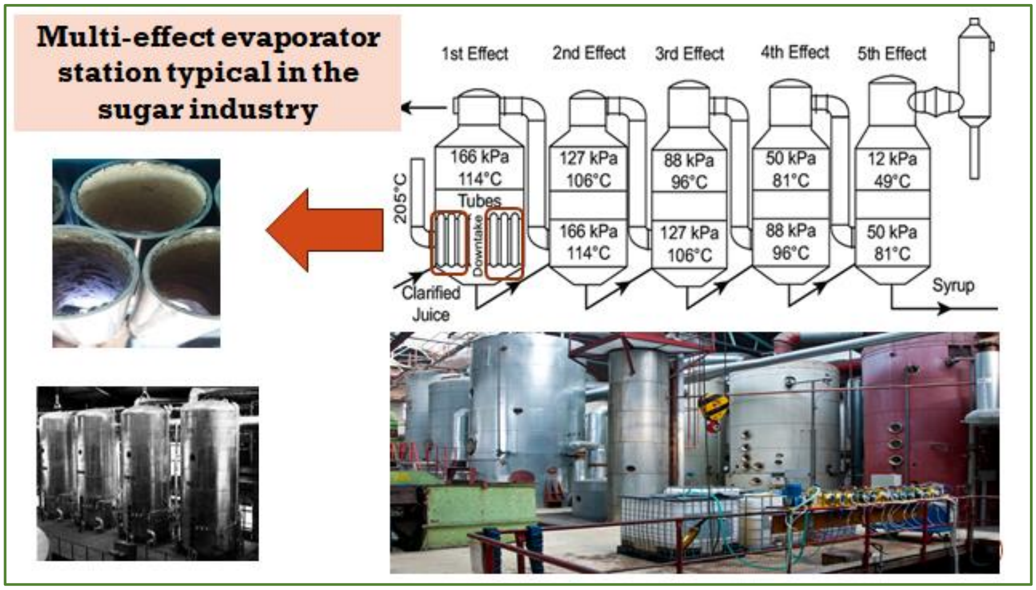

Figure 1: *The multiple effect evaporator system of sugar plant*

Organic acids are " moderate " compounds that breakdown and chelate mineral ions while also disrupting the reprecipitation cycle by isolating and locking up the scale-forming metal ions within a closed-ring structure. Organic acids form by first forming complex surface compounds with the metal in the scale deposit, detaching it from the scale mass and becoming it water-soluble (Chauhan, et al., 2015). There is already a need to produce a variety of novel bio-origin descalant. Sodium gluconate is a biodegradable, odorless, non-corrosive salt that is used as a chelating ingredient in various detergents such as dishwasher tablets to bind to metal ions and ‘soften' the water, enhancing foaming and cleaning effectiveness - notably in hard water. In cooling water systems, gluconate and gluconic acid (Figure 2) are recognized to be efficient non-toxic inhibitors for iron and mild steel (Touir, et al., 2008).

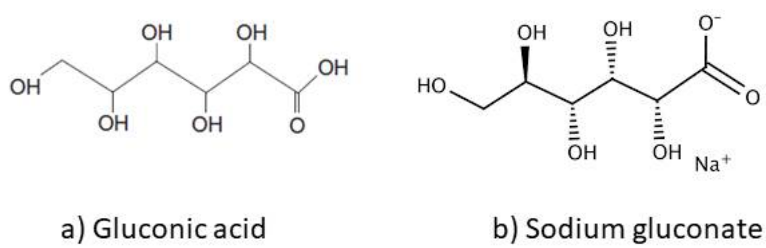

Figure 2: *Chemical formula of the Gluconic acid and sodium gluconate.*

Glutamic acid (Figure 3) is a typical anti-scaling chemical used to control scale deposition (Karar, et al., 2016). Glutamic acid's amino group is protonated (-NH_3^+), while the carboxyl group is dissociated (COO-) in a dipolar state that may act as both a basic (proton acceptor) and an acid (proton donor) and can function as an amphoteric solution (Li, S. et al., 2003). Among the different functional groups present in chelating agents, including hydroxyl, amino, and carboxyl groups, the carboxyl group is recognized as the most effective for providing very stable chelates (Li, N. et al., 2023).

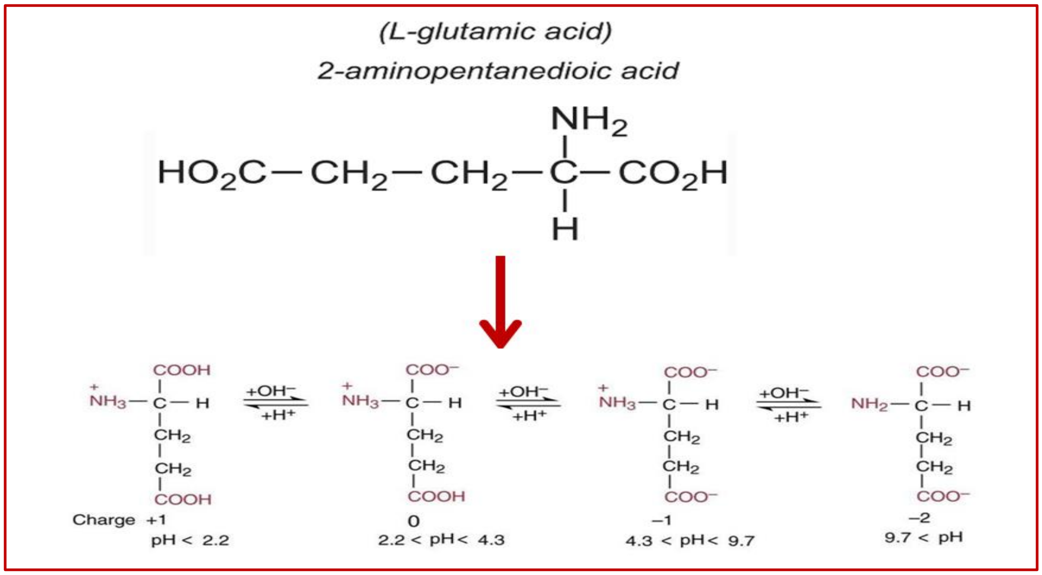

***Figure 3**:Chemical formula of the glutamic acid*

The procedure of removing scale from evaporator bodies is being developed, as are methods of using less expensive mechanical crushing and chemical cleaning agents, as well as methods of lowering the heat transfer coefficient for each evaporation item. The objective of this study was to evaluate and analyses the residues of the evaporator bodies of Quos plant and identify safe, economical chemical cleaning agents and decrease the need of mechanical cleaning methods (mechanical rumen).

2. Materials and Methods

2.1 Chemicals and Reagents

All reagents and chemicals were purchased from ALFA and ELNASR companies in Egypt. The used materials were Sodium hydroxide 1M and HCl 1M. The used materials were Sodium gluconate ($NaC_6H_{11}O_7$) purity 98 % Molar Mass = 218.137 g/ mol and glutamic acid ($C_5H_9NO_4$) Molar Mass= 147.130 g/mol.

2.2 Preparation of scales tubes

Small cut tubes were placed inside large different of quadruple effect MEE system (Multiple Effect Evaporator) during manufacturing of sugar process for two weeks at Quos sugarcane factory (Egypt). Then, the scale tubes were collected to track the effect of the cleaning process as depicted in Figure 4.

Figure 4: *Cleaning of the evaporator tubes with scales*

2.3 Scale Characterization

The deposits inside the sugar plant's evaporative heat exchangers, particularly evaporators 2, 3, and 4, were studied because of their extremely rigid scales. Powder X-ray Diffraction Analysis (XRD, Brucker Axs-D8- Advance Diffractometer, Belgium) was performed at 2Ө range between 10➡-70➡ to analyze the structure of the deposits. Surface morphology and elemental analysis were also examined by SEM and EDX, respectively (Model FEI INSPECT S50) operating at 20 kV.

2.4 Descaling procedures and their effects

2.4.1 Removal of scales

Tubes derived from 1,2,3,4 evaporators were weighted and immersed in different concentration of (1.5 - 10%) from sodium gluconate and glutamic acid molasses at temperature (80 to 100 ºC) with different pH from (4 - 11) for retention time about 4 hours (Figure 5). Calculation of the weight scale differences (W_1-W_2) were recording in the Table. The removal scale was calculated according to equation 2.

$$\% \ \boldsymbol{Scales\ Removal} = \frac{(\boldsymbol{W1-W2})}{\boldsymbol{W_0}} \times \mathbf{100} \ \ \boldsymbol{......Equation\ 1}$$

The cleaning efficiency was then calculated as follows:

where, W_1= weight tube with scale, and W_2= weight tube after cleaning. W_o= weight of total scale (weight of tube with scale – weight of empty tube of scales).

3. Results and discussion

The scale in evaporator units heating systems and boilers is a challenging problem to resolve in many sectors, including sugar, papermaking, and synthetic fertilizer. This allowed for severe economic losses due to its insufficient heat transfer capabilities (Hu, et al., 2006). The creation of a scale is a multistage process that begins with a solution (Chauhan et al., 2015). Because scale adheres so firmly to the walls of tubes and containers, removing it usually necessitates the employment of many procedures. Whatever method is employed, the evaporators units must be paused on sometimes, and evaporators might get worn and corroded (Hu et al., 2006).

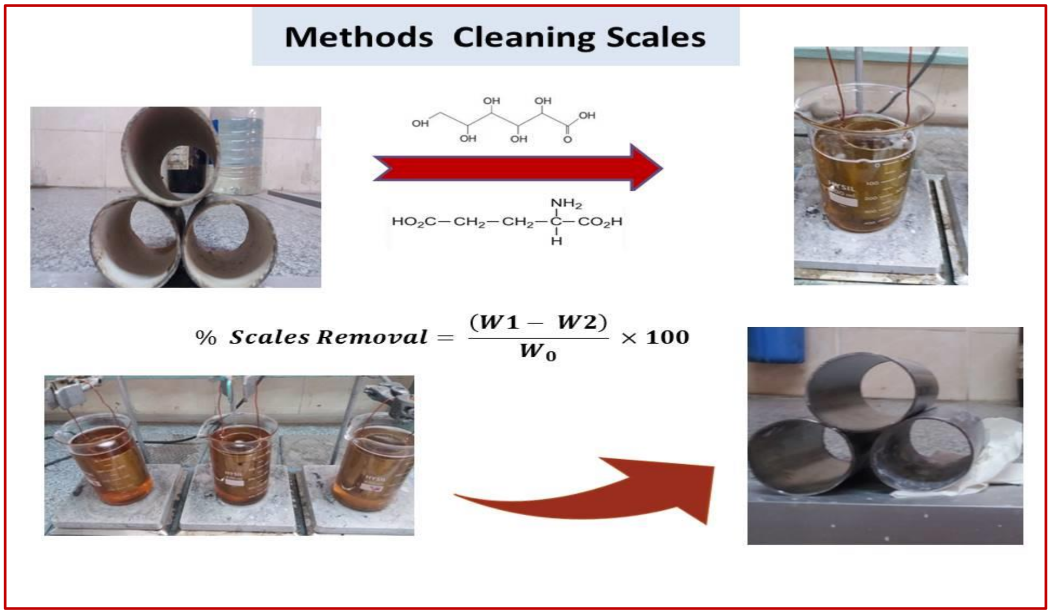

Figure 5*: Evaporator tubes scales in different cleaning solutions*

3.1 Physicals Characterization of Quos Sugar cane Evaporators scales

Some sugar plants were constructed with only four evaporator bodies, such as the Quos factory, where sulphitation treatment [SO_2 +Ca $(OH)_2$] was undertaken. According to the EDX analysis results in Table 1 and Figure 6, the calcium content had the highest metal level, ranging from 12.9 to 17.5% across evaporator EVs (2) and (4). Sulphur S was found in all four evaporative bodies, ranging from 3.9 to 7.2%. Furthermore, Si exhibited a high level of 8.9 % to 14.2 %. While, Mg, Cu, and K have been found in tiny amounts. These observations reported above are comparable to prior ones (Srivastava, et al., 2016). Meal concentrations were always raised in the final evaporator bodies. It was due to a reduction in the water content of the juice from the primary to the forth evaporative body. And the juice content becomes more dense (concentrates) and the impurity elements easily stack in the evaporator's wall tube (Kahsay and Gabbiye, 2015).

Table 1. *EDX analysis for the sugarcane evaporators scales in Quos sugar plant.*

% Element	Evaporator Body 2	Evaporator Body 3	Evaporator Body 4
O	42.24	47.49	45.93
C	27.16	21.37	20.86
Ca	17.52	12.94	16.44
Si	8.96	10.57	14.25
S	3.42	7.24	3.55
Mg	0.37		
Al	0.32	-	0.14
Cu	-	1,97	-
K	-	-	0.25

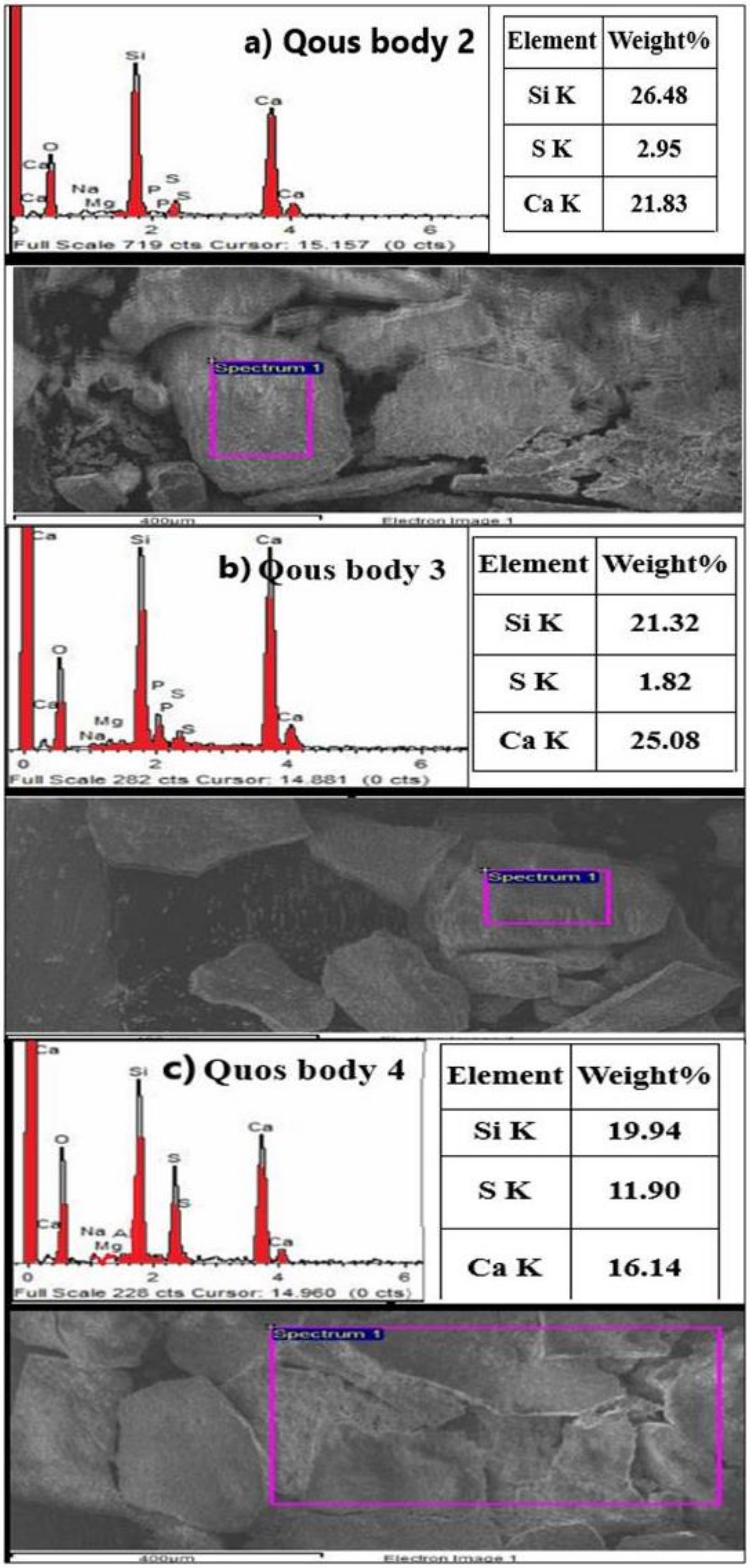

Figure 6:*EDX Scale of evaporators for Quos Sugar plant*

3.1.2 XRD analysis of Evaporation Bodies for Quos factory:

Sulphites and sulphates are the most common scales in sulphitation plant. As shown by XRD patterns in Figure 7, the most confirmed deposits in the evaporator 1 to 4 comprise the bulk of calcium sulphate ($CaSO_4$)- card number (00-002-0134 (D); gypsum ($CaSO_4.2H_2O$)-card number (00-006-0047(D); calcium silicate hydroxide ($Ca_4SiO_5O_{13}.5H2O$) - card number (00-029-0381 (I),Tobemorite ($4CaO.5SiO_2.5H_2O$) card number (00-003-0239 (D) and Wollastonite-2M ($CaSiO_3$) card number (00-027-0088 (C). All evaporative bodies exhibit a resemblance in the peak at 2 theta = 12 with previously known structures for amorphous silica (Bensouissi, Rousse, et al., 2009). Scale can build in sugar juice evaporators through a variety of causes. Any dissolved and suspended particles that are not removed during clarifying might clog the evaporators. Colloidal silica, dirt, plant material, suspended crystals, and colloidal long chain organics are examples of typical particles (East, et al., 2015) (Kulkarni, et al., 1996). Several peaks in the analysis of XRD have greater intensity because their periodicity is larger than that of the other directions. The larger the variation in electron density more is the intensity reflected in the XRD pattern for that plane (Abo, et al., 2021). The greater quantity of compounds including Ca, S, and Si was connected to the process of purifying sugar cane juice. Any residual calcium that remains in the juice after clarification has the potential to contribute to scale formation in the evaporators.

Table 1 : *XRD analysis of Evaporation Bodies of Quos sugar can*

Evaporators No	Composition	Chemical Structure	Card No.
Evaporator 2	Calcium sulfate Gypsum Calcium silicate hydroxide Tobemorite	$CaSO_4$ $Ca\ SO_4 . 2\ H_2O$ $Ca_4SiO_5O_{13}.5H_2O$ $4CaO.5SiO_2.5H_2O$	00-002-0134 (D) 00-006-0047 (D) 00-029-0381 (I) 00-003-0239 (D)
Evaporator 3	Tobemorite	$Ca_5(Si_6O_{16}) (OH)_2$	01-089-6458 (C)
Evaporator 4	Calcium sulfate-gamma Wollastonite-2M	$CaSO_4$ $CaSiO_3$	00-002-0134 (D) 00-027-0088 (C)

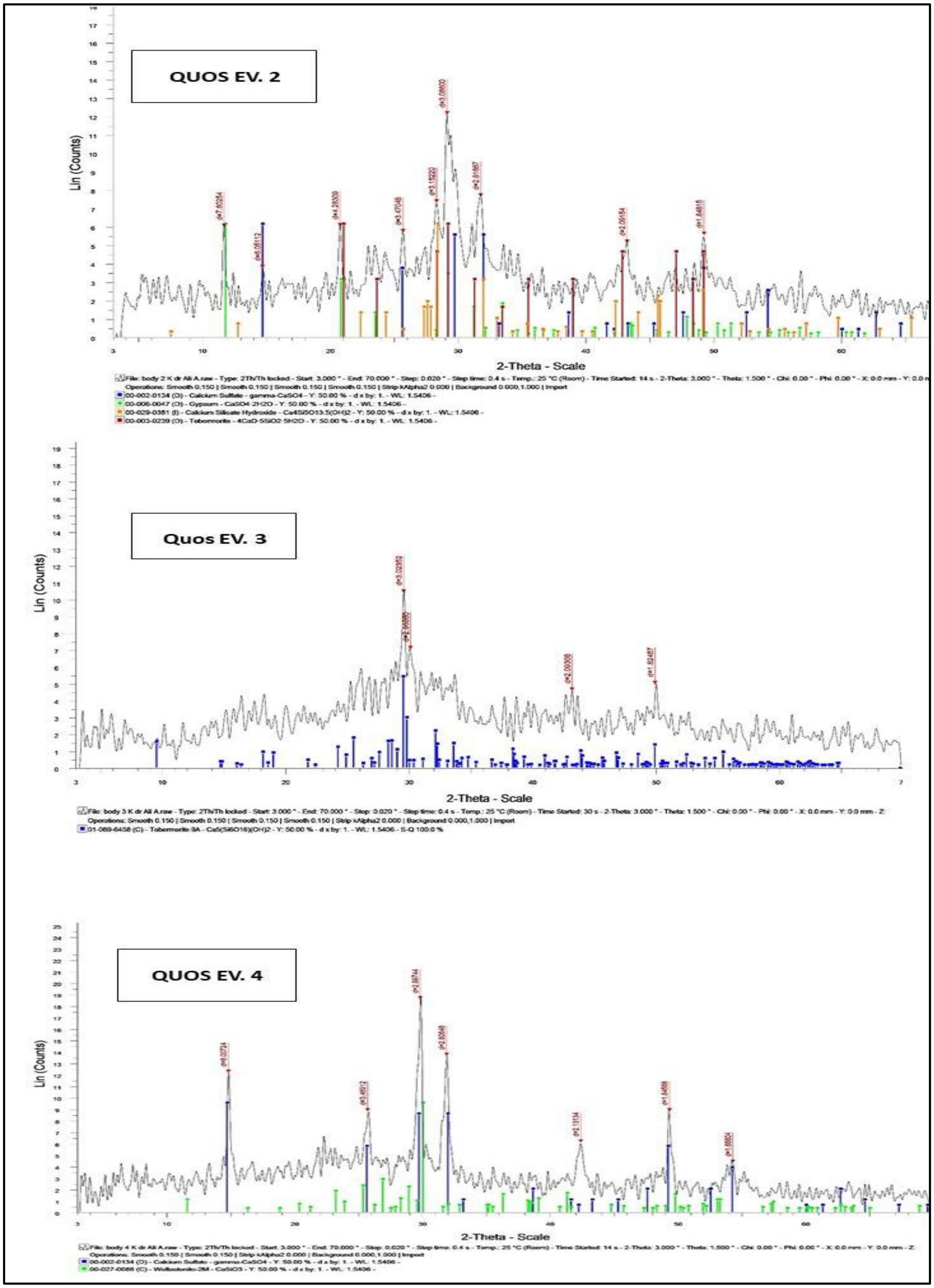

Figure 7: XRD analysis of Evaporation bodies of Quos sugar plant

3.1.3 Scanning Electron Microscopy (SEM)

The external morphology (texture) of the material was examined using a scanning electron microscope (SEM) in relation to its chemical composition and crystalline form. Figures (8) depict SEM images of three Quos sugar factory evaporators. Changes in the morphological properties of the scale in sugar industry evaporator tubes were assessed as a consequence of the effect of employing the clarifying process under ideal circumstances. The scale samples' texture was evaluated, and the scale deposit was categorized as rough. Similar trend were observed by others (Abo et al., 2021). However the concentration of amorphous silica, calcium oxalate, calcium sulphate, and aconitate increases from the first to the last vessel. The solubility of amorphous silica and calcium oxalate decreases with increasing sucrose content and decreasing temperature, resulting in the presence of these scales in the latter (Srivastava, 2011). The SEM photo analysis revealed that the deposit was clearly caused by the calcium component, which was present in greater amounts in the juice than other elements, resulting in the creation of hard scale. The same trend was observed by others (Malik, et al., 2023).

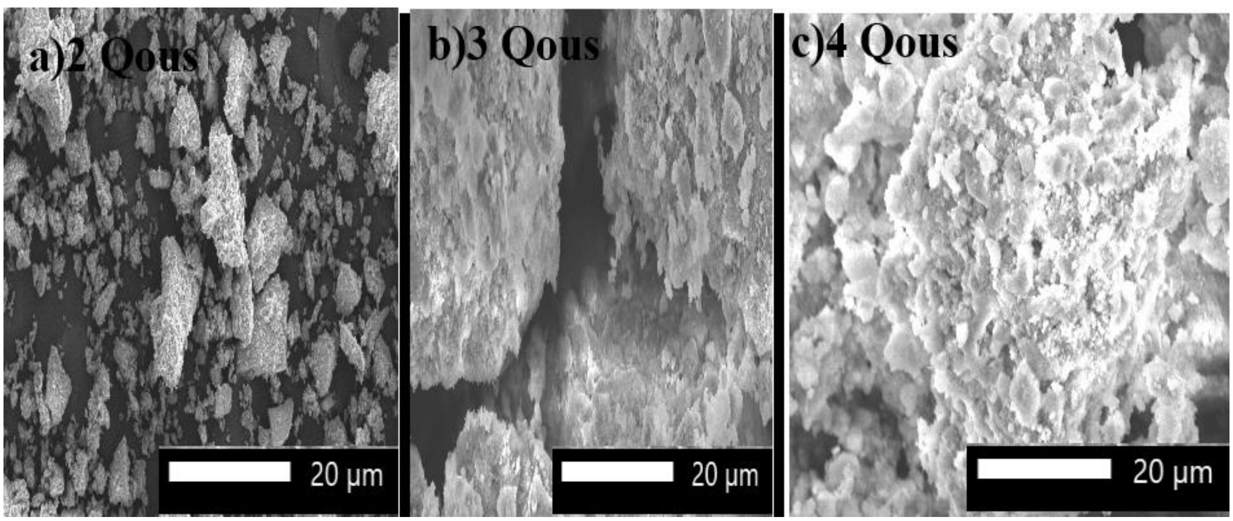

Figure 8: *SEM photo for scale of evaporator bodies (Quos plant)*

3.2 Exploring Sod. Gluconate & glutamic acid for Removal of scales

Chemical methods are particularly strongly significant in removing mineral scales in scale removal operations (Mindler and Epstein, 1986). Chemical cleaning is preferred over mechanical crushing because it cleans big areas quickly and without harming metallic surfaces. Solvents used to remove inorganic deposits usually contain mineral or organic acids, as well as chelating agents. The acid choice is usually dictated by whether the scale is sulphate or carbonate. Another critical consideration is cost-effectiveness and economical. Mineral acids have higher ionization degrees at room temperature than organic acids, which are much weaker and less ionized (Mindler and Epstein, 1986). The effects of sodium gluconate and glutamic acid on scale removal from stainless steel tubes have been examined.

Table 2 displayed that the removal scales in all types of evaporators' bodies do not surpass 41%. Sodium gluconate at a concentration of 5% in the basic medium induced the greatest elimination in the first and fourth bodies. However, the removal effectiveness remains low, particularly in the evaporator 4, which does not surpass 41% (Figure 9). Sodium gluconate cannot be used as a single descaling agent. Sodium gluconate can, however, be used as a stimulating fluid in friendly acid formulas by mixing it with other organic acids (Li, N. et al., 2023).

***Table 2** :Effect of sodium gluconate concentration on the removal of scales*

Concentration	pH	% Removal			
		Body 4	Body 3	Body 2	Body 1
1.5	6.8	39	37.5	36	40
	10	38	38	39	42
3	6.8	28	33	33	28
	10	27	27	27	40
5	6.8	29	28	28	29
	10	41	40	39	41

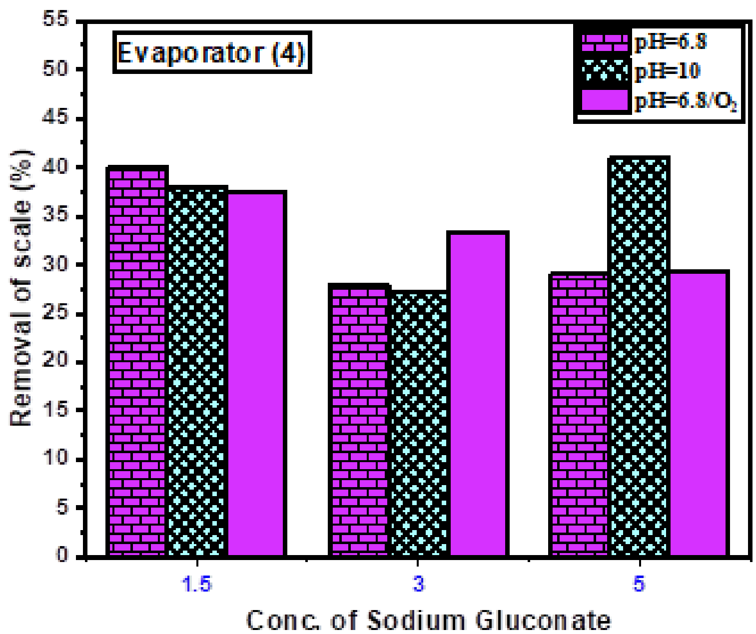

***Figure 9 :**Effect of sodium gluconate concentration on the removal of scales from the evaporative tubs Ev. (4)*

Glutamic acid is a common anti-scaling chemical used for controlling scale accumulation (Karar et al., 2016). Glutamic acid's amino group is protonated (-NH_3^+), while the carboxyl group is dissociated (COO-) in a dipolar state that may act as both a basic (proton acceptor) and an acid (proton donor) and can function as an amphoteric solution (Li, S. et al., 2003). Among the different functional groups present in chelating agents, including hydroxyl, amino, and carboxyl groups, the carboxyl group is recognized as the most effective for providing very stable chelates (Li, N. et al., 2023). The charges on the molecule of L-glutamic acid (Figure 3) are affected by the pH of the solution and its ionization constant (Voges et al., 2017) (Devlin, 2010). Table 3 and Figure 10 demonstrated that glutamic acid's removal effectiveness was great for cleaning the first and second evaporative bodies but insufficient for cleaning the third and fourth evaporative bodies. Hence, the first body includes only organic scale and has an effective removal rate of 83.7%, but the fourth body contains high quantities of silica scale and has an effective removal rate of up to 26.6%.

Table 3: *Effect glutamic acid on the removal of scales from the evaporative bodies (1- 4) at pH= 10, Temp. = 85 °C retention time = 4 hours*

Concentration%	Evaporator no.	% Removal
10	Evaporator Body1	83.7
	Evaporator Body2	50.3
	Evaporator Body 3	36.6
	Evaporator Body 4	26.6

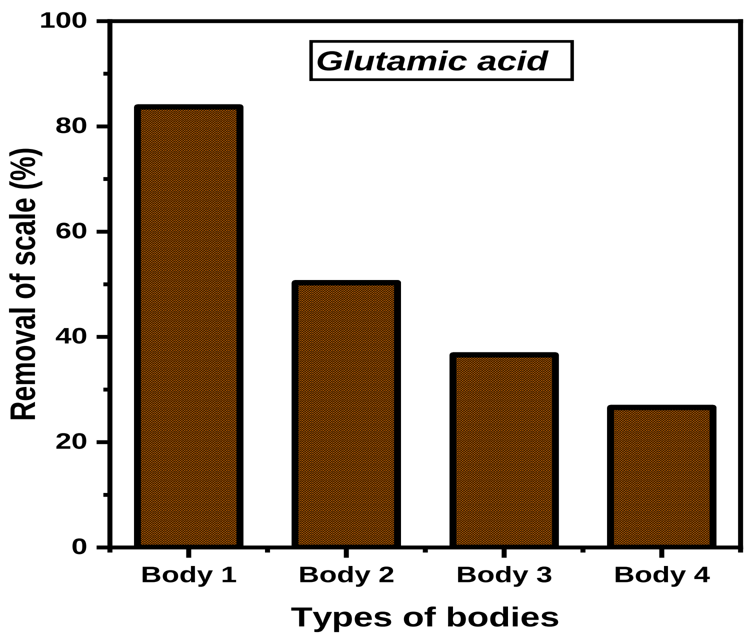

Figure 10: *Study concentration of glutamic acid on the removal of scales from the evaporative tubs bodies*

3.3 Mechanism for scale dissolution by organic acids

Figure 11 displayed the suggested scale dissolution mechanism of glutamic acid and Na-gluconate. The ability of glutamic and glauconitic acids to be utilized as acceptable scale chelating agents to avoid corrosion damage to equipment caused by mineral acid was evaluated to establish their efficacy in removing scale. Organic acids are considered as "mild" acids that can dissolve and chelate mineral ions and break the re-precipitation cycle by isolating and locking-up the scale-forming metal ions within a closed-ring structure. Organic acids function was by initially forming surface complexes with the metal in the scale deposit, detaching it from the scale mass, and thus making it water- soluble (Chauhan et al., 2015).

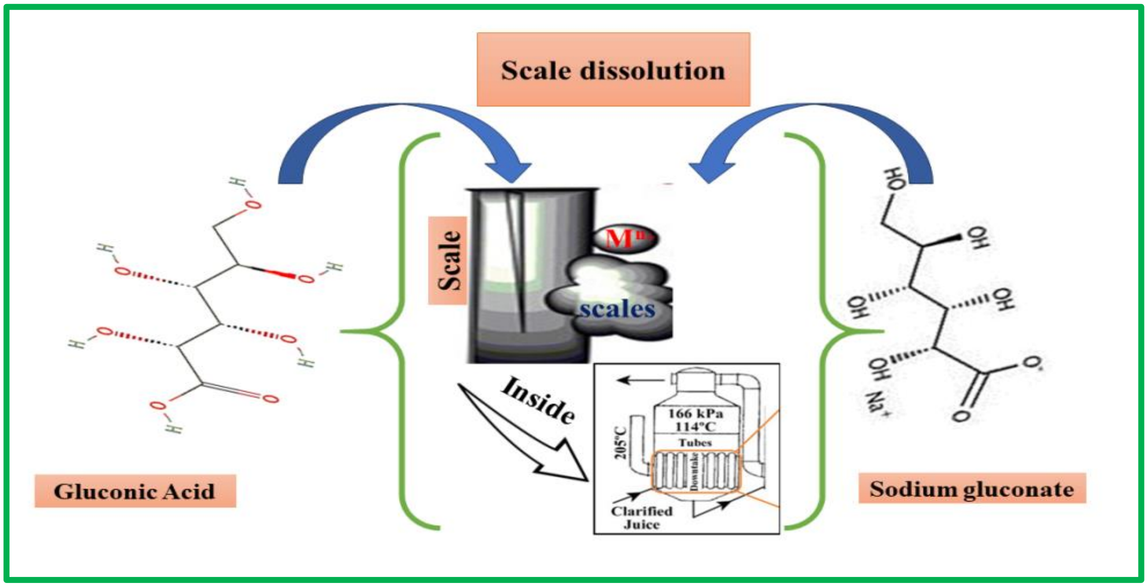

Figure 11: *Mechanism for scale dissolution by organic acids*

Most green chemical inhibitors feature at least one polar group containing a nitrogen, sulphur, or oxygen atom as a chemisorption site. The greater the electron density in the inhibitor structure, the better the inhibitor's efficacy in scale prevention (Kołodyńska, et al., 2008). The Jerusalem artichoke, which is made up of protein, fructose, and glucose (sugar), was found to be an effective scale inhibitor (Ituen, et al., 2017). In other words, the main role of chelates of weak organic amino acid is that added to feed water will be of neutralized form sodium salt. The chelate salt hydrolyzes in the water, releasing organic anion. At coordination sites, the anionic chalet's reactive sites establish chemical interactions with metal cations. For instance, a chelate can interact with iron at six different coordination sites (Chauhan et al., 2015).

4. Conclusions

Despite significant technical advances in recent years, they are still constrained to classic cleaning techniques involving chemical reagents such as NaOH. Sodium gluconate and glutamic acid are used to remove scale from effective evaporators, and it can be affordable at certain points. EDX and XRD methods were used to gain insight into the scale's composition. The predominant scales content in the Quos sugar factories was calcium sulphate due to the sulphitation process used in the clarifying operations. Glutamic acid has a substantially higher removal effectiveness of up to 83% in all types of evaporator bodies, while sodium gluconate only has a removal rate of up to 40%. Green chelating compounds like sodium gluconate and glutamic acid can be used to remove scales from evaporator bodies. This can assist to reduce the need for costly chemical cleaning agents and mechanical crushing, as well as lower the heat transfer coefficient for each evaporator.

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