Characterization of Multiple Effect Evaporators' Scale in New Wonji/Shoa Sugar Factory, Ethiopia

By

Endale Fitamo Arficho

A Thesis Submitted to Department of Applied Chemistry
School of Applied Natural Science

Office of Graduate Studies
Adama Science and Technology University

July, 2020

Adama, Ethiopia

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Advisor: Dr. Dereje Tsegaye

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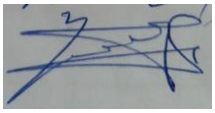
APPROVAL PAGE

We, the undersigned, members of the Board of Examiners of the final open defense by Endale Fitamo have read and evaluated his thesis entitled **"Characterization of Multiple Effect Evaporators' scale in New Wonji/Shoa Sugar Factory, Ethiopia"** and examined the candidate. This is, therefore, to certify that the thesis has been accepted in partial fulfillment of the Degree of Masters in Applied Chemistry.

Name of Student	**Signature**	**Date**
Advisor **Amare A. (PhD)**	**Signature** 	**Date**
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DECLARATION

I hereby declare that this MSc Thesis is my original work and has not been presented for a degree in any other university; all sources of material used for this thesis have been duly acknowledged

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LIST OF ABBREVIATIONS

ACS	American Chemical Society
ANOVA	Analysis Of Variance
ASTM	American Society for Testing and Materials
Bx	Brix
CAS	Chemical Abstract Service
EBT	Erichrome Black T
ECAE	Ethiopian Conformity Assessment Enterprise
ECDSWC	Ethiopian Construction Design and Supervision Works Corporation
EDTA	Ethylene Diamine Tetra acetic Acid
ESC	Ethiopian Sugar Corporation
ES	Ethiopian Standard
ETB	Ethiopian Birr
EU	European Union
EVAP	Evaporator
GSE	Geological Survey of Ethiopia
HTC	Heat Transfer Coefficient
HVA	Handlers Vereeniging Amsterdam
LCSS	Laboratory Chemical Safety Summary
LOI	Loss on Ignition
MEEs	Multiple Effect Evaporators
MW	Mega Watt
NIST	National Institute of Standards and Technology
SD	Standard Deviation
TCD	Tons Cane per Day
TFE	Tetrafluoroethylene (PTFE, Teflon)
WHO	World Health Organization
WSSF	Wonji /Shoa Sugar Factory

ABSTRACT

Scale formation in Multiple Effect Evaporator (MEE) systems is the main unsettled challenge, which is encountered in new Wonji/Shoa sugar factory. Thus, the present study attempted to Characterize Multiple Effect Evaporators' scale in New Wonji/Shoa Sugar Factory, Ethiopia. Micro-Plasma Atomic Emission Spectroscopy (MP-AES) was used to characterize Mg, Si, Fe, and Al. Moreover, Ethiopian Standards (ES 817:2012) was used to determine total solids in clarified juice. Different Standard methods to characterize the clarified water, and evaporators' scale.Minitab Statistical Software, Version 19.1.1 was used for one-way ANOVA test. The Clear juice analysis result revealed that only total solid (19.14 ± 0.32 % w/w) and CaO content (>500 ppm) is high as compared to the standard value (20 -150 mg/L).One-way ANOVA result of CaO versus months revealed that there was very highly significant variation (P < 0.001) between months. The clarified water analysis result was within the standard set by WHO, except the fluoride content (3.57 ± 0.06 mg/L) which is double the European Union (EU) surface water value (1.7 mg/L). The scale collected from new WSSF multiple effect evaporators was analyzed for SiO_2, Fe_2O_3, CaO, MgO, Na_2O, MnO, P_2O_5, TiO_2, H_2O, and Loss on ignition (LOI) using different analytical methods. Except the case for TiO_2 (P > 0.05), all the parameters revealed very highly significant variation (P <0.001) between five evaporators. Moreover, the experimental result of MEEs' scale has shown that SiO_2 and Fe_2O_3 increased from first evaporator to fifth evaporators. CaO, P_2O_5, and LOI of the scale sample are decreasing from first evaporator to the fifth evaporator. The analysis of scale result shown the first evaporators have high content in CaO, P_2O_5, and total volatile substances. From the analysis result, the source of scale is solely from the clear juice obtained from clarification process and is the source of CaO, P_2O_5, and high content of organic matters/volatile substances. Therefore, new WSSF should apply an efficient clarification system primarily to reduce the scale formation.

Key Words: Wonji/Shoa sugar factory, Multiple effect evaporators, Characterization, Fouling, Clarified juice, Clarified water

1. INTRODUCTION

1.1. Background of the Study

Fouling of heat transfer surfaces presents challenges to both designers and operators of heat exchangers in many process industries. Fouling is a process by which deposits settle and accumulate on heat transfer surfaces. Fluids flowing in heat exchangers may contain dissolved substances, suspended matter or may carry substances that promote growth of biological organisms. As a result, deposits may accumulate on a heat transfer surface leading to the formation of a layer. The thermal conductivity of the layer so formed is mostly very low, and therefore, its presence on a heat transfer surface tends to increase the resistance to heat flow. Consequences of fouling in process industries include increased energy consumption, extra maintenance and labor costs, and loss of production opportunities (Mwaba, 2003).

Evaporation of cane juice leads to the accumulation of organic and inorganic scales on the heat exchange surfaces reducing the heat transfer efficiency and the capacity of the evaporator. This increases the juice residence time, which leads to reduced productivity and degradation of the sugar juice.As a result, sugar factories must shut down regularly so that the scale can be removed from the evaporators (Walthew and Turner, 1995; Walthew *et al*., 1997, and Doherty, 2000).These shutdowns are costly because loss of production and the expense of cleaning.

The possible causes of the scaling are impurities in sugar juice or process water (Firdissa, 2012). Scale facilitates the corrosion of surfaces, restricts fluid flow and, because it has low thermal conductivity, its accumulation on metal surfaces hinder heat transfer across the tubes wall. The type of the scale being formed depends on a number of variables such as quality of input materials and intermediate products, flow properties of the fluid, rate of evaporation, and process conditions of the system (Cao, 2010, andBott, 1995).

Evaporator sets in the new Wonji/Shoa sugar factory (WSSF) usually consist of five stages, or effects, in series and two tandems (Tandem A or B). When either set is operational, the other will be made out of operation. The cleaning process involves the use of hazardous chemicals such as sodium hydroxide, sulphamic acid and EDTA, which

create disposal problems for sugar factories. However, in new WSSF case only sodium hydroxide is used for chemical cleaning.

1.2. Statement of the Problem

The new WSSF has been suffering from the after effect of scale formation on multiple effect evaporators (MEEs). It has been observed on the shortage of condensate water for boiler and boiling house processing, low evaporator syrup brix output, low cane crushing due to poor evaporators' performance, high labor and material cost, down time incurred due low capacity of evaporators. It is axiomatic that a necessary step in coping fouling is a thorough understanding of the scale compositions and the mechanisms of formation. Identifying scale components is instrumental to determine which cleaning agent to use and in the design of new cleaning formulations. Evidently, research addressing the issue of scale formation has been reckoned as one of the top priority problems that the Ethiopian sugar corporation would like to contend with in order to make the sugar industries in Ethiopia sustainable. Consequently, the thesis has been designed to fill the gap on the characterization of scale formed, clear juice, and process water. The findings and recommendation of this study can give vital information to Sugar Corporation in general and Wonji Sugar factory in particular on the effects of scale formation on heat exchangers.

1.3. Research Questions

- What has been caused shortage of condensate water for boiler plant?
- What has been caused for low brix of syrup, which passes to the crystallization house?
- What has been affected of low vapor generation from the evaporators for the succeeding process?
- What have been detected as major constituents of scale, clarified juice, and clarified water?

1.4. Objectives of the Study

1.4.1. General objective

- To characterize Multiple Effect Evaporators' scale in New Wonji/Shoa Sugar Factory, Ethiopia.

1.4.2. Specific objectives

- To characterize the scale formed on evaporators' calandria tube.

- To characterize the clear juice obtained from clarification plant that is used an input for evaporators.
- To characterize the clarified water that is used for chemical (Caustic soda) boiling of evaporators.

1.5. Significance of the Study

It had been the top most necessary issue of new WSSF since it was not possible to crush sugar cane to the design capacity due to shortage of condensate water and low performance MEEs. The importance of the study is very crucial, because the factory have been stopped due to low performance of MEEs by tremendous scale accumulation on evaporators calandria tubes. Furthermore, it will demand high steam for juice boiling and this has been resulted in shortage of condensate water for both boiler and sugar processing. Besides, low performance of MEEs have been resulted low output syrup brix (Bx) which would hamper the sugar crystallization process there by low sugar production. The cost of labour, chemical, cleaning materials, maintenance of cleaning materials cost has been high. Therefore, the significance of the study is quite necessary for Sugar Corporation in general and new WSSF in particular.

1.6. Scope and Limitation of the Study

Currently the scale formation on heat exchangers, especially on evaporators has been widely spread all over the sugar industries in Ethiopia like Fincha, Methara, and the newly built sugar factories like Kesem, OmoKuraz #2, OmoKuraz #3, and Tendaho. Moreover, the nature of scale formation may vary depending on the geographical locations, the type of sugar processing technologies, and the sugar cane varieties.

Depending on the budget, time, resource, and the worst condition of new Wonji/Shoa Sugar factory's MEEs the research was conducted only on WSSF's evaporator scale formation.The research study have been done for the assessment of scale formation on MEEs and the characterization of scale, clarified water, and clarified juice was done in depth and in detail. The findings may represent new Wonji/Shoa Sugar factory's MEEs rather than the rest sugar factories in Ethiopia. However, the findings could be used as the base for similar analysis and adopted to other sugar factories.

2. LITERATURE REVIEW

2.1. Mechanisms of Scale Formation

Fouling in the sugar industry is a complex phenomenon involving a number of different deposition mechanisms and fouling species. The first step in understanding fouling in sugar juice heat exchangers (where the majority of the fouling takes place) is by looking at the sugar manufacturing process as a whole. Firstly, the sugar cane is harvested and transported to the factories, where the cane is first shredded and then crushed on the milling train to express the juice. The waste plant material (i.e., bagasse) is used to fire the boilers, creating process steam and electricity. The juice from the milling train is then heated and clarified to remove soluble and insoluble impurities, such as plant material, dirt and scale forming ions (East, 2013).

In evaporators, with cane sugar juice, super saturation is attained due to the removal of water from the juice in the process of its concentration. The driving forces for the formation of deposits on the heat transfer surfaces are the super saturation and the suspended particles present in the juice.

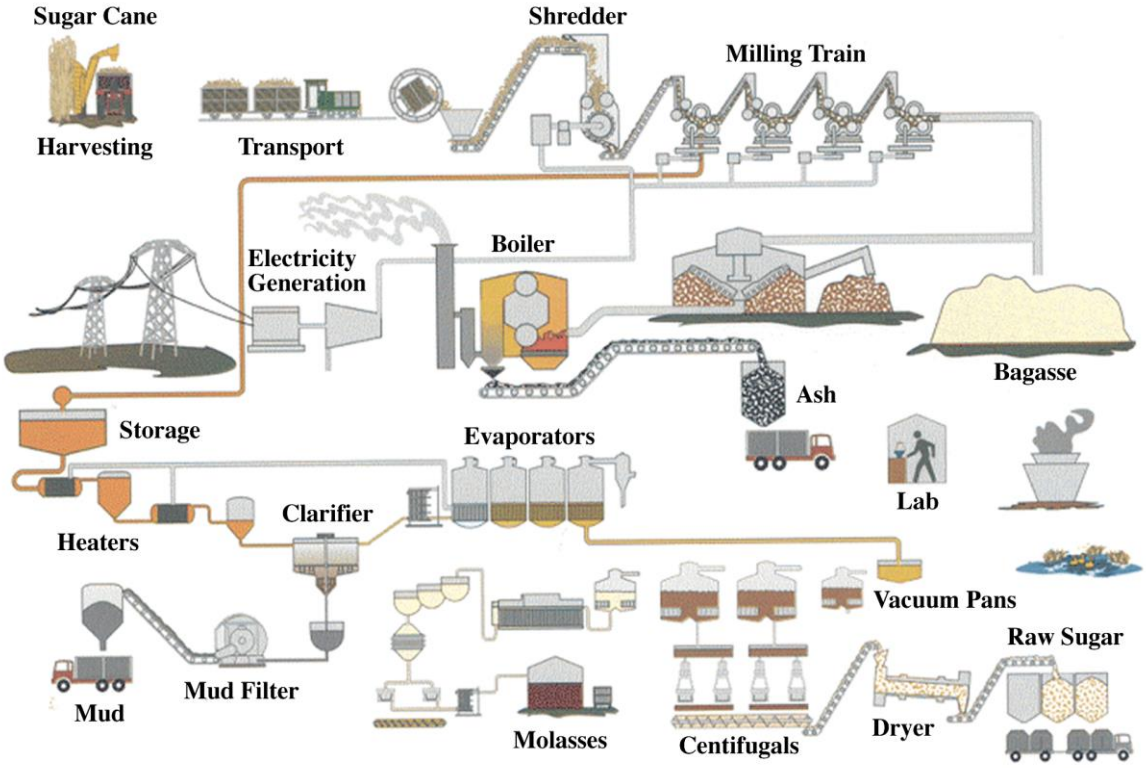

Figure 2.1 The sugar manufacturing process (East, 2013)

Once cane is harvested, it has to enter within 18 – 24 hours to the milling train before significant quality and quantities of sucrose are lost due to microbial activity. After the cane is crushed, the juice is heated to ~ 76 ºC and incubated to remove starch. It is then heated to 100 ºC and clarified (Walford and Walthew, 1996). During clarification, milk of lime ($Ca(OH)_2$) or lime saccharate is added to raise the pH (from ~ 5.4 to ~ 7.0 ± 0.2) and to form calcium phosphate and a sodium poly-acrylate co-polymer is added to aid in the clarification (Walford and Walthew, 1996 and, Doherty and Edye, 1999). Clarification is conducted to increase the quality of the final product and decrease fouling in the evaporators by removing suspended and dissolved solids as well as unwanted organics (e.g., colour precursors) which increases juice quality (Doherty and Edye, 1999). The operating conditions of the evaporators themselves have a large contribution on the scale formation. Flow properties of the solution, the rate of evaporation, and the operating temperature and pressure of the system can all change the rate, type and strength of scale formed (Epstein, 1983). As each factory is different and each region that a factory gets its cane supply is different in terms of cane variety and soil type, the extent and type of scale formation varies from factory to factory, season to season and throughout a season (Doherty, 2000).

During clarification, Milk of lime [Ca (OH)2] is added to assist in the process and for pH control to prevent sucrose inversion. The limed juice is clarified in the clarifier with an anionic flocculent (East, 2013). The clarified juice is then sent to the evaporators and concentrated from ~ 12 to ~ 65 wt % sucrose. The evaporators are where the majority of the fouling occurs.

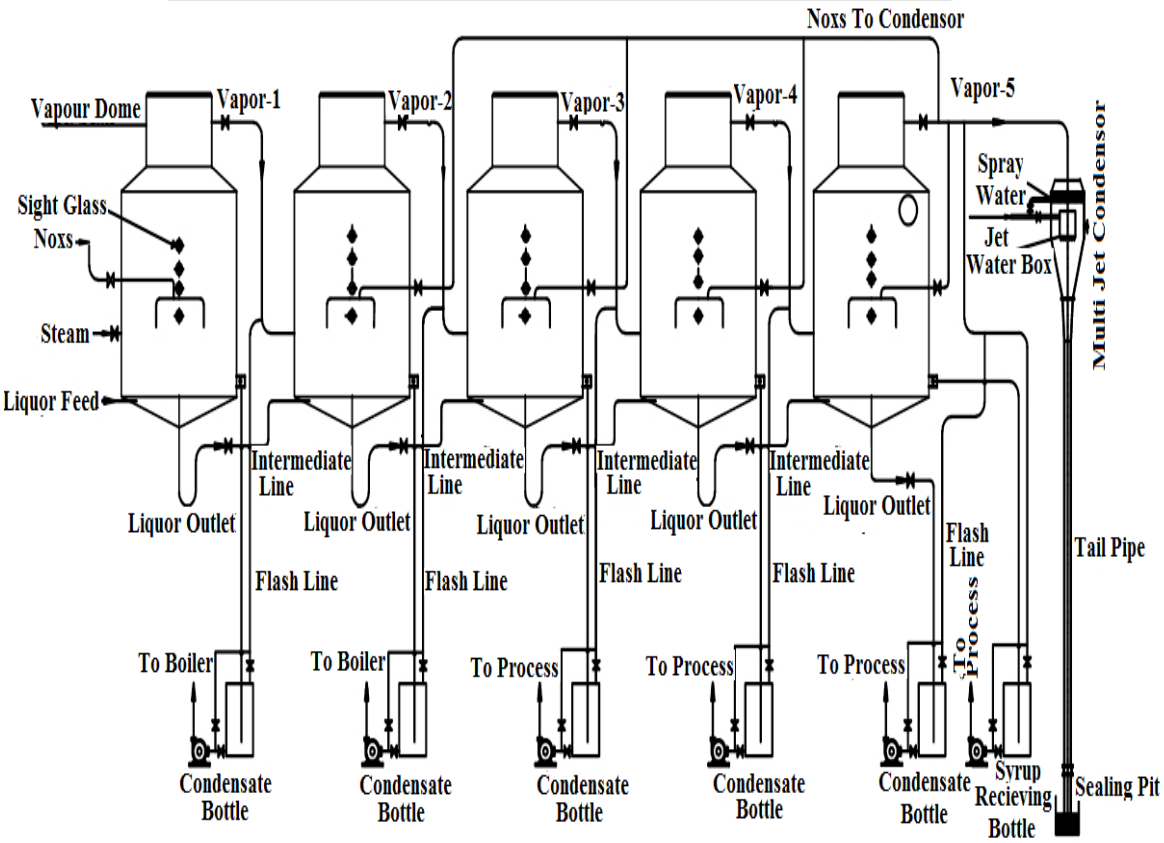

Figure 2.2 Arrangement of Quintuple Effect Evaporator (ESC)

Fouling is a complex process that generally involves a number of physical and chemical phenomena. These phenomena can be grouped into five different mechanisms based on the physical and chemical process essential for each. These mechanisms are crystallization, particulate, chemical, corrosion and biological fouling (Epstein, 1983).

Crystallization fouling takes place when the cane sugar juice becomes supersaturated with the organic and inorganic matter present in it.

Crystallization fouling has two subcategories:

a. **Precipitation Fouling** – crystallization from solution of dissolved substances onto the heat transfer surface, and

b. **Solidification Fouling** – freezing of pure liquids, or crystallization from the melt, onto sub-cooled surfaces.

- Particulate fouling is the accumulation of fine solids suspended in the process fluid onto the heat transfer surface.

- ❖ Chemical reaction fouling is deposit formation at the heat transfer surface by chemical reactions in which the surface material is not a reactant (e.g., polymer production, petroleum refining).
- ❖ Corrosion fouling is the accumulation of products derived from chemical reactions of the surface material on the heat transfer surface.
- ❖ Biological fouling results from the attachment of macro organisms and microorganisms and/or materials generated by these organisms (e.g., adherent slimes).

Deposit formation is not a simple process because a number of fouling mechanisms can occur simultaneously and is further complicated by the variable conditions that occur in industrial systems. It is useful to break deposit formation down into five manageable stages (Epstein, 1983):

A. ***Initiation or induction period*** – Conditions under which a system can foul are established and first layers of foulant are laid, e.g., nuclei for crystallization fouling are formed or organic layers are deposited for biological fouling.

B. ***Transport*** – During this event, deposit-forming components are transported to the surface. The deposition flux $(\dot{m}_d)$ can be related to the concentration gradient by the semi-empirical relationship:

$$\dot{m}_d = k_t(C_b - C_s) \quad \text{..... (2.1)}$$

Where C_b and Cs are the concentrations in the bulk and at the surface respectively and k_t is a transport coefficient. Transport to the surface can occur by convection or diffusion depending on the design and operating conditions of the plant equipment. Some types of diffusion include gravity, turbulent diffusion, diffusiophoresis, Brownian diffusion, electrophoresis and thermophoresis.

C. ***Attachment*** – Attachment of the fouling species to the wall follows transport of the key components to the wall, where the solid that deposits is formed, except in the case of particulate fouling, where attachment is based on sticking probability.

D. ***Removal*** – Removal ($\dot{m}_r$) of the deposit may or may not begin at the same time as deposition and is directly proportional to both the deposit mass (m) and the shear stress (τ_s) on the heat transfer surface, and is inversely proportional to the deposit strength (ψ) which can be expressed by the relationship proposed by Kern and Seaton.

$$\dot{m}_r = \frac{B\tau_s m}{\psi} \quad \text{..... (2.2)}$$

Where *B* is a constant related to the Reynolds number.

E. ***Aging*** – As soon as a deposit is formed it starts to age. Aging processes may include changes in crystal or chemical structure (e.g., dehydration or polymerization respectively). Such changes can increase the strength of a deposit with time. Alternatively, changes in crystal structure, chemical degradation or thermal stresses, may result in a decrease in deposit strength with time. Therefore, a deposit that is not hydro dynamically removable at the beginning of a run may become so when the deposit strength has reached a lower limit, below which it can be removed by moving fluid.

Growth of a fouling layer over time is not necessarily linear but depends on the relationship between deposition and removal that may form one of four relationships shown in Figure 2.1, depending on the dominant fouling event:

(a). Linear growth is typical for scales composed of a pure uncontaminated salt,
e.g., Calcium sulfatedihydrate. Removal of the scale is at a constant rate or nonexistent.

(b). A falling rate curve results from a decreasing deposition rate and/or an increasing removal rate. This occurs when crystallization and particulate fouling occur simultaneously and the scale is with low mechanical strength.

(c). Asymptotic layer growth occurs when the removal rate equals the deposition rate. This can be caused by a change in process conditions or can be inherent in the process itself.

(d). A saw-toothed curve is indicative of a weak scale structure, which leads to the occasional shedding of deposit material. Here the foulant is periodically removed in part or in full by the process.

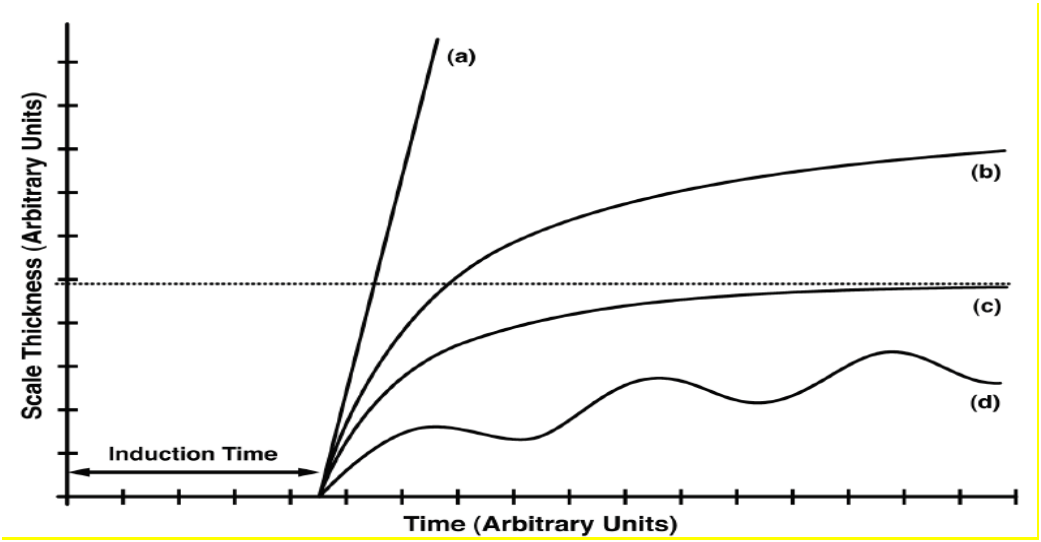

Figure 2.3 Fouling curves (East, 2013)

According to Bruce (2002), deposit that has been accumulated on evaporator surface helps in the growth of scale. Initially, a particular type of fouling takes place and then the other type of fouling starts to dominate. Various investigators (Mwaba, 2003; Yu, 2003; Mwaba*et al*., 2006) have tried to understand the fouling mechanism associated with the sugar cane industry. Crystallization fouling is the dominant fouling mechanism in the cane sugar industry (Mwaba, 2003). As to Nandagopal and Ramamurthy (1976) and Walthew (1996), major constituents of scale forming materials are silicates, phosphates, sulfates, carbonates and calcium, whereas minor constituents are oxalates, magnesium, iron and aluminum oxide.

Conversely, Yu and Sheikholeslami (2009) suggested that composite fouling is the main mechanism of fouling in cane sugar evaporators. Composite fouling is a form of fouling that involves more than one type of fouling species or mechanism (Sheikholeslami and Ng, 2001; Yu, 2003). These different fouling species interact with each other to produce a final deposit. The interactions between calcium carbonate scale and silica particles and observed that silica deposition reduces the mean calcite crystal size formed on the heat transfer surface (Andritsos and Karabelas, 1999). Moreover, calcium oxalate monohydrate and amorphous silica forms tenacious composite scale in later effects of multiple effect evaporator (MEE) system processing cane sugar juice (Yu *et al*., 2003). Crees *et al*. (1992) suggested that hydroxyapatite and gypsum are the main foulants in sugar industries. Kahsay and Gabbiye (2015) and Gurram and Menkhaus (2013) found $CaSO_4$ as the main foulant in sugar industries. Further, Kahsay and Gabbiye (2015) treated the cane sugar juice with H_2SO_4 to reduce $CaSO_4$, whereas Gurram and Menkhaus (2013) proposed sulfate ion removal and enzyme recovery in reducing the cleaning cost. East *et al*. (2013) simulated the formation of calcium oxalate and SiO_2 composite in MEE systems used in sugar industries.

Results from the scale analysis and juice composition showed that the deposits are formed via different fouling mechanisms dependent upon the temperature, concentration and location in the process. Hydroxyapatite is most likely to be the first scale component deposited on the tube surface. Compared to hydroxyapatite, there is a lower driving force for the deposition of calcium oxalate and it is deposited on cooler surfaces when the $[Ca^{2+}]$ and $[C_2O_4^{2-}]$ are high enough for it to be thermodynamically favorable. On the coolest surfaces with the highest sugar concentrations amorphous SiO_2 is the dominant scale

component, probably due to the aggregation of colloidal SiO_2 which will be encouraged by the high ionic strength of the environment (East, 2013).

Figure 2.4 The inside of evaporator covered with scale (East, 2013)

2.2. Heat Transfer Coefficients (and Rates of Heat Transfer)

The heat transfer coefficient (HTC) is a measure of the efficiency of the transfer of heat from one medium to another. It includes a temperature differential, surface area and heat transfer dependence.The advantage of using the heat transfer coefficient as opposed to an evaporation rate per unit area is the fact that it takes into account temperature difference so is a good basis for comparison between effects where operating conditions such as temperature may vary. In general, the rate of evaporation purely on a mass flow basis can be improved by increasing the saturated temperature of the condensing steam, but this does not necessarily increase the heat transfer efficiency.

2.3. Scaling and its Effect

Scale formation is of interest to the sugar industry and many areas of industry where heat transfer and flows are affected by the formation of this scale. The scale decreases the heat transfer rates in heating media and can cause severely depleted flow through piping if it forms thick scale layers in certain industries. In the sugar industry, this scale needs to be

removed from heat transfer surfaces to allow the rate of heat transfer to achieve required throughputs. This cleaning means downtime for mills and costs a significant amount of money in chemicals and lost production. Since the scaling causes a reduction in heat transfer efficiency, equipment typically is oversized to account for a certain amount of scaling over time; this increases capital costs of equipment. The scale can form from a number of scale-forming minerals that are inversely soluble; that is the solubility of the mineral decreases with increasing temperature.

Minerals like this include calcium and silicon minerals. Common scale components for water include calcium carbonate, calcium sulfate, calcium phosphate, silica, magnesium silicate, iron-based scales, zinc phosphate and zinc hydroxide. In water systems scale, inhibition methods include softening with lime or lime/soda, acid treating, scale inhibitors and dispersants as well as surfactants. Factors affecting scaling rates include concentration, pH, flow velocity and temperature. This is a broad and general classification and this will need to be refined to decide on model parameters specific to the sugar industry and the evaporator set under consideration. In both the sugar and many other industries, pH control is key to reducing scale formation.

2.4. Chemical cleaning of evaporators

While dealing with the hard scales of evaporators, the mechanical cleaning as such has provided to be rather incompetent for satisfactory scale removal and for better heat exchange. To mark it, it is essential to know the general composition of scales and the behavior of their components towards various cleaning agents employed.

Scales in the evaporators of cane sugar factories consist always of inorganic and organic non-sugars. The main cation in scales is calcium with small amounts of magnesium combined with sulphates, phosphates, organates, silicates and sesquioxides. The scales in the first bodies of the evaporators are higher in phosphates and organic matter and can be easily removed either by chemical or mechanical means of cleaning due to their relatively soft nature. Last body scales are usually of sulphates, sulphites, silicates and sesquioxides like Fe_2O_3 and Al_2O_3. These are compact and hard in nature. Magnesium when compared to calcium relatively is in higher concentration in scales of the first effects, than of the last effects.

Chemical cleaning consists of the removal of the incrusting compounds from the scaled surface with chemicals either alkaline or acidic in nature. For a complete removal of incrustation, it is not necessary to remove or dissolve all the scaling components. If only a considerable part of the scaling components is dissolved, the practical result is that the coherence of the incrustation is last the scale disintegrates and can be flushed off with a stream of water or by brushing. Hence, selective chemical is employed to suit the purpose.

2.4.1. Alkali Treatment

Among alkalis, which have been recommended in recent years for cleaning the evaporator surface is solution of caustic soda. The most recommended and efficient one is the caustic soda solution of 25 – 30 ^{0}Bx. Containing 10% soda ash and 5 – 10% of common salt. With this combination, the effect of caustic soda is more pronounced in cleaning the scales of the first bodies of evaporators. Scales of first bodies of evaporators are rich in organic matter. As far as the organic matter is concerned, the best and cheapest way of decomposition is by hydrolysis. The caustic soda solution hydrolyses the organic components of the scales.

NaOH + Organic matter ----------→ Sodium Organate

The sulphates and sulphites of the scales as such can be disintegrated by the action of a concentrated solution of sodium hydroxide.

$CaSO_4 + 2NaOH$ ----------→ $Na_2SO_4 + Ca(OH)_2$

The soda ash added to the NaOH solution during the process of soda boiling has the added advantage in the removal of the sulphates and sulphites of the scale by transforming them into simple carbonates that are soluble.

$CaSO_4 + Na_2CO_3 + H_2O$ ----------→ $Na_2SO_4 + CaCO_3 + H_2O$

$CaSO_3 + Na_2CO_3 + H_2O$ ----------→ $Na_2SO_3 + CaCO_3 + H_2O$

The silica is not dissolved by an acid or alkali other than hydrofluoric acid. However, to a very small extent, NaOH of higher concentration on prolonged heating forms a soluble silicate.

$SiO_2 + 2NaOH$ ----------→ $Na_2SiO_3.H_2O$ =Soluble sodium silicate

2.4.2. Fluoride Treatment

The most important factor in the cleaning treatment is the removal of silica, the hardest incrustation component of the scales. Basing on the fact that sodium/ammonium bifluoride will disintegrate the structure of the silicate scales by the dissolution of SiO_2, now a day, the bifluoride are being used along with alkalis or acid to the extent of 1.5 – 3.0% over

HCl/alkali. However, the biflourides are at their best when used only with hydrochloric acid. It is because of the reason that the fluoride liberate hydrofluoric acid on the action of HCl and the resulting hydrofluoric acid dissociates silica.

When the bifluorides are used along with alkali, through the reactions are very slow, a partial dissolution of silicates takes place.

$6NH_4HF_2 + 2SiO_2 \text{ ----------} \rightarrow 2\ H_2SiF_6 + 6NH_3 + 4H_2O$

$3NaHF_2 + SiO_2 \text{ ----------} \rightarrow Na_2SiF_6 + NaOH + H_2O$

Between these two bifluorides, the reaction with ammonium bifluoride appears to be faster than that with sodium bifluoride. This may be because ammonium bifluoride in aqueous solution at boiling temperature undergoes a partial dissociation into

$NH_4HF_2 + H_2O \text{ ----------} \rightarrow NH_4OH + 2HF$

The hydrofluoric acid thus liberated reacts with silica

$6HF + SiO_2 \text{ ----------} \rightarrow H_2SiF_6 + 2H_2O$

In addition, the silica in the scales is dissolved and the structure is disintegrated. Hence, it is more advantageous to use ammonium bifluoride than sodium bifluoride along with soda solution.

If silica in scales is present in the form of calcium silicate, $CaSiO_3$, the NaOH at high temperatures will decompose it into sodium silicate, which is water-soluble.

$2NaOH + CaSiO_3 \text{ ----------} \rightarrow Na_2SiO_3 + Ca(OH)_2$

However, mostly the silica is present in the form of SiO_2 only to the maximum extent in the last body scales. During the process of soda boiling, NaOH reacts with the organic matter and hydrolyses it and partial dissolution of silicate takes place at higher concentration. During these reactions, the sodium hydroxide is consumed and the concentration goes down. Nevertheless, the NaCl used along with the soda solution regenerates a part of NaOH and thus used at its best to raise the solution strength. These are chain reactions to a certain extent until all the chemicals are consumed.

$2NaOH + CaSO_4 \text{ -------} \rightarrow Na_2SO_4 + Ca(OH)_2$

$CaSO_4 + Na_2CO_2 + H_2O \text{ --------} \rightarrow Na_2SO_4 + Ca(OH)_2 + CO_2$

$2NaCl + Ca(OH)_2 \text{ -------} \rightarrow CaCl_2 + 2NaOH$

The efficiency of the chemical cleaning depends upon the concentration of the chemicals used. The quantity to be used is determined by the intensity of scaling and the composition of the scale. In case of very hard scales of silica, content above 25% the caustic soda treatment followed by acid treatment will prove effective. Even though it is claimed that after chemical cleaning the scales can be washed off easily, practical experience shows that at least a mechanical brush cleaning is necessary to follow with.

3. MATERIALS AND METHODS

3.1. Description of the Study Area

The study was conducted at new (Wonji/Shoa Sugar Factory) WSSF multiple effect evaporators, which is located in rift valley of east Shoa zone of Oromia region near Adama city and 110 to 120 Km away from Addis Ababa, Ethiopia. It is located between 8˚31′ N and 39˚12′E at an altitude of 1565 meter above sea level (midland) and the average annual rainfall is about 800mm as shown in Fig, 3.1 and 3.2.

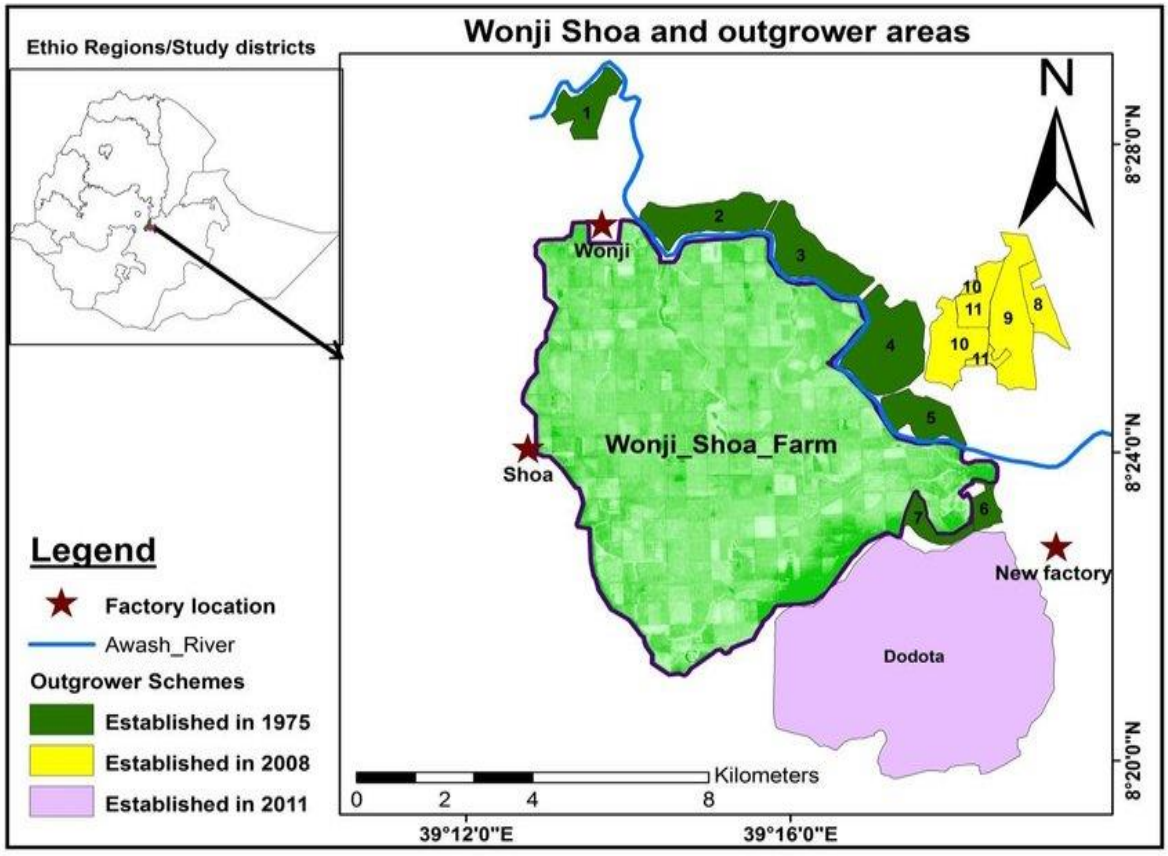

Figure 3.1 Map of the study area (MengistuWondimu, 2015)

The two old sugar factories Wonji and Shoa were established in 1954 and 1962 E.C. respectively. Then, have stopped production since July 2012 and July 2013, respectively. The two factories constructed by the Holland Company known as Handlers Vereeniging Amsterdam (HVA) had a capacity of producing 75 thousand tons of sugar a year. The sugarcane plantation land of these two factories was 7,000 hectares out of which 1,000 had been planted by out growers (ESC).

The two oldest factories with a new and modern one, an expansion project had been carried out both in the cane cultivation field and the factory since 2005/2006. Moreover, the factory expansion project has come into its completion in July 2013. A new Wonji/Shoa Sugar Factory (WSSF) is established at Dodota some 21Km from Wonji. The Factory is located in the rift valley, Oromia region, in East Central Ethiopia, some 149 Km South East of Addis Ababa at an elevation of 1540 above sea level. The newly built and modern WSSF has currently a design capacity of crushing 6,250 Tons of cane per day (TCD) and producing 174,946 tons of sugar per annum, which with further expansion work will reach up to 12,500 TCD maximizing its production to 220,700 tons of sugar a year. The Factory is capable of producing 31Mega watt (MW) electricity and out of this 7 to 8 MW is used for its electricity demand and the rest is to be exported to national grid (ESC).

Figure 3.2 New Wonji/Shoa sugar factory (Photo)

3.2. Materials and Chemicals

3.2.1 Apparatus and materials

Flame photometer (Model 410, classic, Sherwood Scientific Limited,UK), Spectrophotometer, for use at 510nm, providing a light path of 1cm, Acid-washed glassware, Ion meter, A spectrophotometer(Model 6715 Cole-Parmer Ltd.Beacon Road, Stone, Staffordshire, ST15 0SA, UK) suitable for measurements at 660 or 880 nm with a light path of 1 cm, Water bath (95°C), electric muffle with an automatic temperature controller,Agilent 4200 MP-AES, Agilent technologies, USA, analytical balance (Mettler AE 100, Hightstown, NJ, USA), Analytical balance, accurate to 0.1 mg, Calibrated spindle, Brix cylinder.

3.2.2 Chemicals and reagents

Standard potassium solution(NIST KNO_3, Certipur ®,USA)Standard sodium solution(NIST $NaNO_3$, Certipur ®,USA), HCl (37 % , Hisea ,China) with less than 0.00005% iron, Hydroxylamine solution (H_3NOH, Aldrich-43227); Ammonium acetate buffer solution($C_2H_7NO_2$, LCSS); Sodium acetate solution (CH_3COONa-,NIS,USA);Phenanthroline solution(ACS reagent, 99%,$C_{12}H_8N_2$);Standard iron solutions(NIST Fe, USA);Phenolphthalein indicator (Hln/PhPh,$C_{20}H_{14}O_4$),Methyl orange indicator($C_{14}H_{14}N_3NaO_3S$,Merck Millipore,ACS), Sodium carbonate solution(Na_2CO_3, Pacific Northwest National Laboratory, Richland, WA,USA) Potassium dichromate indicator($K_2Cr_2O_7$,India) Silver nitrate solution, $BaCl_2$, ASTM type II water (deionized water), Na_2CO_3, Standard sulphate solution (NIST Na_2SO_4, Certipur ®, USA) ; Standard fluoride solution (NIST NaF, Certipur® USA) ; Fluoride buffer;Molybdate - antimony potassium tartrate solution (ACS reagent, ≥ 90 % Sigma –Aldrich), distilled water, Ascorbic acid solution($C_6H_8O_6$, LCSS),Sulfuric acid (98 % H_2SO_4, Sigma-Aldrich, ACS), Sodium bisulfite ($NaHSO_3$) solution, Ammonium persulfate (98.5% CAS, China), Standard phosphorus solution (TraceCERT®, Sigma-Aldrich); 50% H_2SO_4, 48% HF, NH_4Cl, macerated filter paper, Bromine water, methyl red, bromthymol blue indicator, NH_4OH, 2%NH_4Cl; Potassiumpyrosulphate, 1% Potassium permanganate solution, 6% Cupferron solution, 10% HCl; Stannous chloride, Potassium di chromate, Mercuric chloride, mixed acid (50-50 H_2SO_4 and H_3PO_4), Barium diphenylamine sulphonate, and Potassium dichromate, ammonium hydroxide, standardized 0.1% ammonium oxalate solution, ceric sulphate, diammonium phosphate, $CaCO_3$, sodium carbonate, 5% HCl, ammonium nitrate, ammonium citromolybdate, 5 % HNO_3(ACS Grade, Merck),EDTA

solution, Ammonia, Lead acetate: Horne's dry lead, Potassium Ferro cyanide (Merk quality) powder, EBT indicator.

3.3. Characterization of Clarified water

A representative three clarified water samples that collected at different time of processing new Wonji/Shoa sugar factory was collected from clarified water inlet line to MEEs used for chemical boiling were analyzed. Moreover, Ethiopian Construction Design and Supervision Works Corporation (ECDSWC) did determination of different Cations and Anions with different test methods and an average of the result was taken.

3.3.1. Determination of Na+ and K+ using Flame Photometer

Procedure

Potassium determination:

Flame photometer was adjusted for selecting proper photocell, wavelength, slit width, fuel gas and air pressure, steps for warm up, correcting for interference and flame background, rinsing of burner, sample ignition and emission intensity. A blank and potassium calibration standards were prepared, in the range of0-100 mg K/L. Instrument was set zero with standard containing no potassium. Emission at 766.5 nm was measured and calibration curve was prepared and sample was run in the same way as the blank and standards. Potassium concentration of the sample was determined from the curve.

Sodium determination:

The same steps for flame photometer adjustment was followed as potassium determination.A blank and sodium calibration standards were prepared, in the range of0 - 100 mg Na/L. Instrument was set zero with standard containing no sodium. Emission at 589nm was measured and calibration curve was prepared and sample was run in the same way as the blank and standards. Sodium concentration of the sample was determined from the curve.

3.3.2. Determination of total iron using 1, 10 – Phenanthroline

Procedure

50 mL of mixed sample was taken into a 125 mL conical flask. 2 mL Conc HCl 1 mL NH_2OH. HCl solution, a few glass beads was added and heated to boiling until the volume is reduced to 20 mL, cooled, and transferred to a 50 mL volumetric flask. 10 mL

$NH_4C_2H_3O_2$ buffer solution and 4 mL Phenanthroline solution was added diluted to the mark with water. Mixed and allowed 10-15 min. for colour development. All standards and sample; Photometer absorbance readings at 510nm was taken.

3.3.3. Determination of Chloride by Mohr Argentometric Precipitation Titration

Procedure

Clarified water sample was filtered to remove any suspended materials. 50mL of filtrate sample was pipetted out in large porcelain disc. 3-4 drops of phenolphthalein indicator was added a pink color was disappeared. Red colour was obtained after methyl orange is added and N/50 sodium carbonate solution was added until thecolour of the solution changes to orange. The resultant solution was transferred in a 250 mL conical flask and 1mL of potassium chromate indicator was added. Then, standard N/50 silver nitrate solution slowly added from the burette with constant shaking. A white precipitate of silver chloride was obtained. Addition process was continued slowly and a red colour appeared in the flask, which was disappeared on shaking. Then, silver nitrate solution was added drop by drop until a permanent reddish brown colour was obtained.

3.3.4. Determination of sulphate using Turbidimetry

Procedure

Formation of barium sulphate turbidity:

100mL-clarified water was placed into a 250 mL Erlenmeyer flask. 5mL conditioning reagent was exactly added. It was mixed in a stirring apparatus. During stirring, a spoonful of $BaCl_2$ crystals was added and timing was begun immediately. It was exactly stirred for 1-min at constant speed.

Measurement of barium sulphate turbidity:

After the stirring time had ended, the solution was poured into absorbance cell. Turbidity was measured at 30 sec. intervals for 4 min. In addition, maximum reading obtained in the 4 min. was recorded.

3.3.5. Determination of Fluoride using Ion- selective electrode

Procedure

A series of working standards were prepared by diluting 5.0, 10.0 and 20.0 mL of standard solution to 100 mL, corresponding to 0.5, 1.0 and 2 mg F^- /L, respectively. 10 to 25 mL standards were taken and clarified water in 100 mL beakers. The samples and the standards were brought to the room temperature and an equal volume of buffer was added to each

beaker. Manufacturer's instructions were followed to set up and the ion meter was calibrated using standards in the prescribed range. Stirring before immersing electrodes avoided so as not to entrap air bubbles. A direct reading from instrument was taken.

3.3.6. Determination of Phosphate using Ascorbic acid, Molybdate blue

Procedure

50mL clarified water was transferred into a 125 mL Erlenmeyer flask and 1 mL of 11 N sulfuric acid was added. 0.4 g ammonium persulfate was added, mixed, and boiled gently for approximately 30-40 minutes or until a final volume of about, 10 mL was reached. It was heated for 30 minutes in an autoclave at 121°C (15-20 psi). Then, it was cooled, diluted to approximately 40 mL and filtered. Sodium bisulfite was added, mixed, and placed in a 95°C water bath for 30 minutes (20 minutes after the temperature of the sample reaches 95°C). Then, it was cooled and diluted to 50 mL. 1 mL of 11 N sulfuric acid and 4 mL of ammonium molybdate-antimony potassium tartrate was added to 50mLdistilled water and mixed. 2 mL of ascorbic acid solution was added and mixed. After 5 minutes, the absorbance at 650 nm with a spectrophotometer was measured. The phosphorus concentration was determined from the standard curve. A 5 cm cell was used.

3.3.7. Determination of Total Hardness

Procedure

The clarified water first filtered and 50 mL of the filtered clarified water sample pipette into a 250 mL Erlenmeyer flask. After adding about 2 mL of buffer solution, 3-4 drop of Erichrome black T indicator solution added. After the appearance of wine red color, the solution is titrated with 0.05 M EDTA from the burette until a blue end point is reached by the disappearance of the last purple coloration.

$$\textbf{Calcium determination (Ca)} = \textbf{titer} \times \frac{\textbf{250 cm}^3 \times \textbf{Calcium (mg)} \times \textbf{100g}}{\textbf{50g} \times \textbf{10cm}^3 \times \textbf{1000mL}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (3.1)$$

Total hardness (as ppm $CaCO_3$)=Titration volume (mL) × 20

Calculation for Molarity of EDTA: $M \times V\ (MgSO_4) = M' \times V'$ (EDTA)

3.4. Characterization of MEEs' scale

Methodology

Sampling

The representative MEEs' scale sample was collected from each evaporators during descaling processing. The samples had been collected within fifteen days' time interval when one of the two MEEs set was out of operation for the sake of cleaning the calandria tubes and it is then collected with plastic bags.

Sample preparation

A representative sample of 2Kg for each evaporator from the bulk sample was taken. It was grounded by mortars and pestles to pass a 60-mesh sieve. The hard and soft portion of the sample that were segregated during the grinding and sieving are remixed to homogenize. Then, the separate portion of the sample was taken for complete silicate analysis to determine major and minor oxides by the laboratory of Geological survey of Ethiopia (GSE).

3.4.1. Determination of Loss on ignition (LOI)

Procedure

1g of scale sample was weighed into a clean, tared, platinum crucible and it was dried for an hour at 140^0C in an oven and reweighed. It was ignited to 1000^0C in an electric muffle. It was cooled in a desiccator, weighed, and calculated.

$$\frac{[(140^0\text{C Wt.}) - (1000^0\text{C Wt.})] \times 100}{\text{Sample Wt. at } 140^0\text{C}} = \% \text{ LOI} \quad \text{----------(3.2)}$$

3.4.2. Determination of Fe_2O_3, Al_2O_3, MnO_2, TiO_2

Procedure

5mL of water and 25 – 35 drops of 50% H_2SO_4 was added to a crucible containing the sample obtained from LOI determination and the crucible was filled with 48% HF. It was heated from the room temperature to 110 ^{0}C and evaporated to apparent dryness. The heat was slowly increased to 230 ^{O}C and the fuming H_2SO_4 was ceased. It was placed on Meeker burner and high temperature was held for 30 – 40 minutes. It was then transferred to 400 mL beaker and 200 – 250 mL distilled water, several drops of bromine water, 5 g of NH4Cl, 2 g of macerated filter paper, and 1 – 2 ml of HCl was added. The beaker was placed on hot place and digested for one hour. After removing from heat few drops of

methyl red, and bromthymol indicators was added. NH_4OH dropwise was added to a pH of 5.5. The end point was checked with pHydrion indicator paper. The solution was boiled for 1 – 3 minutes and filtered through Whattman # 41 H filter paper. The precipitate was washed with boiling 2 % NH4Cl. The filtrate was reserved for CaO and MgO determination. The drained precipitate was placed in the original platinum crucible. It was dried and ignited at $400^{O}C$ in an electric muffle to remove filter paper. The temperature was slowly increased to $1200^{O}C$ and maintained for 30 – 40 minutes. It was cooled in a desiccator and weighed as RO_2 and R_2O_3. (Where**R**= either **Mn** or **Ti**, and $\mathbf{R_2}$ = either **Fe** or **Al**)

$$\frac{\text{[(Wt. ignited ppt. + Crucible) – (Crucible wt.)]}}{\text{Dry wt. Scale sample}} = \% \text{ total } RO_2 + R_2O_3 \text{ ---------} \quad (3.3)$$

3.4.3. Determination of Iron, Titanium, Aluminum Oxides (Cupferron precipitation)

Procedure

10 g of potassium pyrosulphate was added to the precipitate in the platinum crucible. It was fused over a low flame of a Meeker burner until complete solution of the precipitate is effected. The fusion was cooled and placed in a clean 250-mL beaker. The crucible was washed with boiling water and the washes was added to the beaker. And, it was filled with water until 150 – 200 mL including the washes. 10 mL of 50% H_2SO_4 was added and heated carefully with stirring to complete solution. It was filtered quantitatively through Munktells "0" filter paper into a 400-mL beaker. Filter paper was washed five times with boiling water. 30 mL of 50% H_2SO_4 was added, diluted to 300 mL and cooled to $5^{0}C$. Macerated filter paper and 1% potassium permanganate solution was added dropwise until a faint but persistent pink color is attained. It was stirred in a 6% cupferron in a small increments and 10 – 30mL of solution was added to complete the precipitation. The solution was filtered through double paper composed of Whattman #40 supported by # 41 H. It was filtered on a suction apparatus and 1-2 mL of Cupferron reagent was placed on the suction flask. The precipitate was washed 15 – 20 times with small portion of cold 10% HCl with 1-2mL Cupferron solution per liter. The filtrate was discarded. The precipitate was placed on tared porcelain crucible and dried, heated slowly in an electric muffle and was completed the ignition at $1000^{0}C$ for one hour. It was cooled, weighed, and recorded as $Fe_2O_3 + RO_2(TiO_2, V_2O_5, ZrO_2)$.

$$\frac{[(Fe_2O_3 + RO_2) - Fe_2O_3] \times 100}{\text{Dry wt. of scale sample}} = \%(Ti0_2 + V_2O_5 + ZrO_2) \text{ ---------- (3.4)}$$

3.4.4. Determination of ferric oxide (Fe_2O_3)

Procedure

RO_2 and R_2O_3 precipitate was gently fused with approximately 10 g of potassium pyrosulphate over a Meeker burner until solution of the residue is complete. The fusion was allowed to cool and the fusion button was placed in a clean 250mL beaker. The crucible was washed with boiling water and the total volume of water was kept to 100 – 150 mL including the washes. 10 – 15mL of HCl was added on the sample and heated carefully over a Bunsen burner until the fusion cake is in solution. Sufficient stannous chloride was added drop wise until the yellow color of the solution disappears. The sample was placed in the refrigerator to cool. When the sample was cooled below the room temperature, it was titrated with standardized potassium dichromate solution. 35mL of saturated mercuric chloride solution was rapidly added and stirred. It was let to stand 1-3 minutes until the solution turns milky. 15 mL of mixed acid (50-50 H_2SO_4 and H_3PO_4) and 8-10 drops of iron indicator (Barium diphenylamine sulphonate) was added and it was titrated with standard potassium dichromate. After the end point changed to very deep purple the burette readings before and after titration was recorded and calculated as Fe_2O_3.

$$\frac{\text{mL of dichromate used} \times \text{titer of dichromate} \times 100}{\text{Dry wt of sale sample}} = \% \text{ total } Fe_2O_3 \text{ ---------- (3.5)}$$

3.4.5. Determination of CaO (Oxalate precipitation)

Procedure

The combined R_2O_3 filtrate was evaporated to 100 – 150 mL and was passed through the Munktells “0” filter paper into 400mL beaker. 5-10 mL HCl and 5g of ammonium oxalate was added. The solution was heated on hot plate to approximately 80^0C. Calcium oxalate was precipitated by the dropwise addition of ammonium hydroxide to a bright yellow color with methyl red indicator. The solution and precipitate was allowed to stand for one hour. It was filtered through the Munktells “0” filter paper into a 600mL beaker. The precipitate was washed off the filter paper into the original beaker and it was repeated the precipitation using 1g of ammonium oxalate. The filtrate was reserved. The second

precipitation was let to stand for two hours and filtered. The precipitate was washed five times with 0.1% ammonium oxalate solution. The combined filtrates and washes were reserved for MgO determination. The sulfuric acid solution of calcium oxalate was heated to boiling and titrated to a faint but permanent yellow with standard ceric sulfate. A blank was run to determine the amount of ceric sulphate.

$$\frac{\text{(mL ceric sulfate – mL blank)} \times \text{Titer} \times 100}{\text{Dry wt of scale sample}} = \%\ \text{CaO} \quad \text{---------- (3.6)}$$

3.4.6. Determination of MgO (diammonium phosphate precipitation)

Procedure

The combined filtrate of calcium oxalate precipitation was evaporated to dryness. 35-50 mL of HNO_3 was added and covered with ribbed watch glasses and let to dryness. 35 mL of HCl was added and the solution was taken to dryness. It was cooled and 50mL of 5% HCl was added to dissolve the residue. It was transferred quantitatively to a clean 250mL beaker up to 150mL. 2.5 g of diammonium phosphate and several drops of bromthymol blue indicator was added. Ammonium hydroxide was added dropwise until a precipitate of magnesium ammonium phosphate begins to form. Ammonium hydroxide was slowly added until the solution is deep blue in color. The precipitate was allowed to settle for a few minutes to an hour. Then an excess of 5 mL ammonium hydroxide per 100 mL was added. The solution was covered with a watch glass and placed in the refrigerator for a minimum of four hours or overnight. The precipitate was filtered through Munktells "0" filter paper. The original beaker was placed under the filter. The precipitate was dissolved and washed through filter paper with 5% HCl. 0.1g diammonium phosphate and bromthymol blue indicator was added for the second precipitation. It was filtered through the filter paper and washed with 5% ammonium hydroxide. The precipitate and filter paper were placed in a tared, porcelain crucible; ignited to 1100^0C holding at 400^0C to allow for the combustion of the filter paper. It was cooled, weighed and calculated as MgO.

$$\frac{\text{Wt. of magnesium pyrophosphate} \times 0.3621 \times 100}{\text{Dry wt. of scale sample}} = \%\ \text{MgO} \quad \text{---------- (3.7)}$$

3.4.7. Determination of silica (SiO_2) (HCl dehydration)

Procedure

Approximately 5g of scale was weighed into a tared, platinum crucible. It was dried at 140^0C, Cooled in a desiccator and the dry sample weight was recorded. 1g of $CaCO_3$was added to the sample in the platinum crucible. 6 g of sodium carbonate was added and stirred with a glass rod until a uniform mixture is obtained. The fusion mixture was covered by the thin layer of sodium carbonate. The crucible was covered with a platinum lid and placed in an electric muffle furnace. After slowly increasing the temperature was held at 1000^0C for one hour. The fusion was heated until all bubbles had been evolved and the resultant mixture was quiet and molten. The sample was allowed to cool. The cake was tapped out from crucible into a 250 mL casserole. Water was added to cover the fusion button.

It was covered with a large watch glass. 5% HCl was filled into the crucible until the remnant of the cake was loosen. This solution and any solids was quantitatively added to the casserole. After solution of fusion button, the casserole was placed on a hot plate. The cover glass was rinsed and removed and the solution was evaporated to dryness approximately for 15 hours. The casserole containing the desiccated silica was cooled. 25 mL of HCl was added under the cover glass. At low temperature, it was refluxed on the hot plate for 5 – 10 minutes. 90-100 mL of boiling water was added and stirred until most of sodium chloride and other soluble salts are in solution. The solution was filtered through Whattman # 40 filter paper. The silica was washed ten times with boiling 5% HCl and then five times with boiling water. The residue and the paper was placed on the original platinum crucible. It was ignited in an electric muffle holding at 400^0C until combustion was completed. Then it was preceded to 1200^0C and maintained this temperature for 30 minutes. It was removed, cooled in a desiccator, weighed, and recorded the weight as crude silica. 5 mL of water and 10 drops of 50% H_2SO_4 was added to the crucible and it was filled with 48% HF. It was placed on a hot plate and the temperature was slowly increased to 110^0C. It was evaporated until all fumes of sulfuric acid were removed. It was placed on the Meeker burner and covered with a platinum lid, heat cautiously at first, and slowly increased the heat until the maximum of the burner was attained and continued for one hour. It was removed from heat, cooled in a desiccator, and weighed. The heating and weighing steps were repeated until the constant weight was obtained. The weight was recorded as fume silica residue. The percent SiO_2 was calculated.

Crude SiO_2 – Silica residue = net wt. SiO_2

Net wt, of SiO_2 × 100= % SiO_2 ---------- (3.8)
Dry wt. of scale sample

3.4.8. Determination of Phosphate (P_2O_5)

Procedure

3g of scale was weighed into a clean 250mL beaker. It was covered and 40mL of water and 15 mL of HNO_3 was added. It was digested near boiling for an hour and brought to boiling. It was cooled, filtered through Munktells "OK" filter paper and washed with nitric acid-ammonium nitrate solution in small amounts to keep the total volume of 75mL. And, the residue was discarded. 100mL of ammonium citromolybdate reagent was added to the filtrate and brought to boil with caution and was let to stand overnight. Using vacuum it was filtered through tared, fritted crucible. It was washed five times with nitric acid-ammonium nitrate wash solution and then washed twice with water. It was removed from vacuum and washed the bottom of crucible with water. It was dried at 105^0C for 1-2 hours, cooled, weighed, and calculated as P_2O_5.

Wt. Phosphorous complex × 0.0378 × 100= % P_2O_5 ---------- (3.9)
Wt. of scale sample

3.5. Characterization of Clear juice

Methodology

Sampling

Clear juice (CJ) sample of 1-liter was collected from the clarification plant during the operation time; that is when there is different cane variety had been crushed and during steady state operation. Moreover, clear juice pH was kept within the range 6.9 – 7.2 and temperature was within the range 85 – 95^0C. The bulk sample had been collected at three days' time interval for fifteen days of each set of MEE. The sample was taken to Addis Ababa for analysis at the laboratory of Ethiopian Conformity Assessment Enterprise, ECAE.

3.5.1. Determination of Mg, Fe, Al, Si

Instrumentation

All measurements were performed using an Agilent 4200 (Micro Plasma Atomic Emission Spectroscopy) MP-AES, with nitrogen supplied from an Agilent 4107 Nitrogen Generator. The sample introduction system consisted of a double pass spray chamber and OneNeb nebulizer. The instrument was controlled by the powerful and easy-to-use MP Expert software. The MP-AES features continuous wavelength coverage and MP Expert features an extensive wavelength database that allows the selection of wavelengths that are appropriate for the concentration range required for the analysis.

Table 3.1 4200 MP - AES Operating Parameters

Parameter	**Value**			
Element	Mg	Fe	Al	Si
Wavelength	280.271nm	259.940nm	396.152nm	251.611nm
Nebulizer	OneNeb			
Nebulizer flow rate	Default (0.75L/min)			
Spray chamber	Double pass glass cyclonic			
Pump rate	15 rpm			
Auto sampler	Agilent SPS 3			
Read time	1 Second			
Number of replicates	3			
Sample uptake delay	30 Seconds			
Rinse time	40 Seconds			
Stabilization time	20 Seconds			
Back ground correction	Auto			
Gas source	Agilent 4107 Nitrogen generator			

Procedure

Standards and sample preparation

The clear juice (CJ) was diluted 20× with 5% HNO_3(ACS Grade, Merck). No other modifiers or suppressants were required.

Standards were prepared from a 10,000 mg/L multi element standard (Inorganic Ventures). All calibration blanks and standards were prepared in 5% HNO_3.

3.5.2. Determination of total solids

Procedure

Aluminum weighing dishes were pre-dried by placing them in a 105 ± 3ºC drying oven for a minimum of four hours. The dishes were cooled in a desiccator. Using gloves or tweezers to handle the dishes, a pre-dried dish was weighed to the nearest 0.1 mg. This weight was recorded. The CJ sample was thoroughly mixed and then weighed out an appropriate amount to the nearest 0.1 mg, into the weighing dish. CJ samples should be passed through a 0.2 µm filter prior to analysis. The weight of CJ sample plus weighing dish was recorded. Each CJ sample was analyzed in duplicate, at minimum. The CJ sample was placed into a convection oven at 105 ± 3^{O}C for a minimum of four hours. The CJ sample was removed from the oven and it was allowed to cool to room temperature in a desiccator. The dish containing the oven-dried sample was weighed to the nearest 0.1mg and this weight was weighed recorded. The CJ sample was placed back into a convection oven at 105 ± 3^{O}C and it was dried to constant weight. Constant weight was defined as ± 0.1% change in the weight percent solids upon one hour of re-heating the sample.

3.5.3. Determination of CaO in clarified juice

Procedure

About 300 mL CJ sample was taken obtained from the factory.150 mL of the CJ was transferred into a conical flask. The solution was clarified by lead acetate. About 60 mL of clarified solution was transferred into a dry conical flask. Powdered potassium Ferro cyanide was added little by little until not further precipitate forms. It was thoroughly shaked and filtered. The filtrate was tested for the absence of lead with potassium iodide. The lead free filtrate was collected in a conical flask. 10 mL of this filtrate was pipetted out into a clean conical flask previously rinsed with distilled water and dried. 3-4 mL of ammonia solution was added and a few drops (4-5) of EBT indicator solution was added till a pink red color appears. It was titrated against EDTA solution. The end point was indicated by sharp change of color from red to blue. The volume of the titrant was noted.

Observed (Uncorrected brix) = B

Titration reading = V ml

$$\mathbf{CaO = V \times 100\ mg/L}$$

$$\mathbf{= \frac{V \times 100 \times 100}{B}\ mg/L\ per\ 100\ Bx} \text{ ---------- } (3.10)$$

4. RESULT AND DISCUSSION

4.1. Characterization of Clarified Water

Table 4.1 presents results of clarified water used for chemical boilingfor descaling of MEEs of new WSSF. The result indicate every values are within WHO's maximum allowable concentration, except the fluoride concentration (3.57 ± 0.06 mg/L) which is doubled the EU Directives surface water regulations (1989) concentration (1.7 mg/L). Moreover, the total hardness of the clarified water was analyzed to be 39.17 ± 7.98 mg/L as compared to the standard value 0 – 300 mg/L. So therefore, the clarified water has no contribution on scale formation on evaporators. Ataklti Kahsay and Nigus Gabbiye (2015) have investigated the input water quality analysis of Wonji-Shoa sugar plant and water quality parameters including total hardness, TDS, COD, BOD and EC were measured. Moreover, the result was below the standard values so they concluded that the impurities of cations and anions have not any influences for scale/bio-fouling conditions in the evaporation plant.

Table 4.1 Clarified water sample of new WSSF analysis result

Tests	**Test results** **Average±SD (mg/L)**	**Test methods**	**WHO maximum allowable conc. (ppm)**
Sodium (mg/L Na^{+})	71.33 ± 4.72	Flame Photometry	200.0
Potassium (mg/L K^{+})	16.0 ± 3.46	Flame Photometry	-
Total Iron (mg/L Fe^{2+}& Fe^{3+})	0.68 ± 0.14	Spectrophotometry using 1,10-Phenothroline	0.3
Chloride (mg/L Cl^{-})	38.27 ± 1.19	Mohr Argentometric titrimetry	250.0
Sulphate(mg/L SO^{2-}_{4})	29.34 ± 2.00	Turbidimetry	400.0
Fluoride (mg/L F^{-})	3.57 ± 0.06	Ion- Selective Electrode	1.7 *
Phosphate (mg/L PO^{3-}_{4} -P)	0.16 ± 0.012	Spectrophotometry using Ascorbic acid, Molybdate blue	-
Total Hardness	39.17 ± 7.98	Titrimetry using EDTA	0 - 300

* EU Directives surface water regulations (1989) result.

4.2. Characterization of MEEs' Scale

Scales in the evaporators of cane sugar factories consist always of inorganic and organic non-sugars. The main cation in scales is calcium with small amounts of magnesium

combined with sulphates, phosphates, organates, silicates and sesquioxides. The scales in the first bodies of the evaporators are higher in phosphates and organic matter and can be easily removed either by chemical or mechanical means of cleaning due to their relatively soft nature. Last body scales are usually of sulphates, sulphites, silicates and sesquioxides like Fe_2O_3 and Al_2O_3. These are compact and hard in nature.

Table 4.2 presents the percentage of major and minor oxides result of scale taken from new Wonji/Shoa sugar factory MEEs and analyzed for SiO_2, Fe_2O_3, CaO, MgO, Na_2O, MnO, P_2O_5, TiO_2, H_2O, and Loss on ignition (LOI). The result indicates that SiO_2 and Fe_2O_3, is on an increasing order from the first to the fifth evaporator (i.e. SiO_2 is 3.28 ± 0.014 % of Evap. # 1 and 16.02 ± 0.007% of Evap. # 5, and Fe_2O_3 is 1.66 ± 0.012 % of Evap. # 1 and 38.54 ± 0.013% of Evap. # 5). This is primarily due to the fact that the solubility of silica/silicate as SiO_2 are known to decrease with increase in sucrose concentration leading to the predominant presence of these in deposit components in the last evaporators of MEE where concentrations of sugar are substantially high. One of the sesquioxides, Fe_2O_3 is in higher concentration in the scale of last evaporators. Moreover, the CaO and P_2O_5 higher in the first evaporators (i.e. CaO is % 25.33 ± 0.011 % of Evap. # 1 and 13.10 ± 0.014 % of Evap. # 5, and P_2O_5 is 2.07 ± 0.011% of Evap.# 1 and 0.72 ± 0.011% of Evap. # 5). This is due to high precipitation of calcium phosphates at higher temperature of first evaporators, which is removable either by chemical or mechanical means of cleaning. The LOI is high in the first evaporators, i.e. 60.70 ± 0.013 % of Evap. # 1 and 31.28 ± 0.03% of Evap. # 5, indicating that high amount of organic matter deposition on the first evaporators at high temperature and are easily removable.

One – way ANOVA result revealed that TiO_2 their P-values was 0.285 (i.e. $P > 0.05$),so therefore there is no significant variation between the five evaporators. However, the P-values for SiO_2,Fe_2O_3, CaO, MgO, Na_2O, MnO, P_2O_5, H_2O, and LOI and the P- value is < 0.001 and it is highly significant variation between the five evaporators (See appendix).

Table 4.2 Complete scale analysis result (%) of MEEs' of new WSSF

Scale sample	SiO_2	Fe_2O_3	CaO	MgO	Na_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
Evap#1	3.28 ± 0.014^{d}	1.66± 0.012^{e}	25.33± 0.011^{a}	0.10± 0.014^{d}	2.16± 0.01^{a}	0.14± 0.02^{d}	2.07± 0.011^{a}	0.01± 0.012^{a}	2.54± 0.013^{e}	60.70 ± 0.013^{e}
Evap#2	4.24 ± 0.013^{c}	18.96± 0.015^{d}	24.50± 0.013^{b}	0.48± 0.013^{b}	0.18± 0.02^{c}	0.30± 0.01^{c}	1.11± 0.012^{b}	0.02± 0.011^{a}	2.88± 0.01^{d}	47.78 ± 0.02^{d}
Evap#3	13.72 ± 0.014^{b}	23.48± 0.014^{c}	19.40± 0.012^{c}	0.82± 0.014^{a}	0.06± 0.01^{d}	0.32± 0.01^{bc}	0.47± 0.013^{e}	0.04± 0.013^{a}	3.63± 0.011^{b}	38.27 ± 0.01^{b}
Evap#4	-	29.92± 0.014^{b}	14.00± 0.010^{d}	0.78± 0.011^{a}	0.20± 0.01^{c}	0.36± 0.02^{b}	0.58± 0.014^{d}	0.04± 0.014^{a}	4.62± 0.012^{a}	33.61 ± 0.021^{a}
Evap#5	16.02 ± 0.007^{a}	38.54± 0.013^{a}	13.10± 0.014^{e}	0.42± 0.014^{c}	0.48± 0.02^{b}	0.44± 0.01^{a}	0.72± 0.011^{c}	0.03± 0.013^{a}	3.39± 0.01^{c}	31.28 ± 0.03^{c}

* Mean ± SD that do not share a letter are significantly different.

4.3. Characterization of Clear Juice

The working concentration range of the standard solutions is summarized in Table 4.3.

Table 4.3 Working concentration range of the 4200 MP - AES standard solutions

Element	4200 MP-AES concentration range (mg/L)	Correlation Coefficient
Mg 280.271nm	0 – 1	0.99988
Fe 259.940 nm	0 – 0.5	0.99960
Al 396.152 nm	0 – 1	0.99978
Si 251.611 nm	0 – 0.5	0.99995

Table 4.4shows the concentration and recovery results of the four elements in the clear juice. The recovery results for Mg, Fe, Al, and Si in the clear juice using this method were

within +/- 10% of the assigned value. All results measured in this study were within the certified ranges.

Table 4.4 Recovery results of four elements in the clear juice using the 4200 MP - AES with the nitrogen generator

Clear juice	**Certified Value (mg/L)**		**Found (mg/L)**	**% Recovery**
	Assigned value	**Range**		
Magnesium	0.05	0.01 – 0.09	0.036 ± 0.22	102
Iron	-	-	-	-
Aluminum	0.15	0.1 – 0.25	0.1 ± 0.36	107
Silicon	0.02	0.01 – 0.06	0.018 ± 0.24	99

Long term stability

A sample clear juice solution (diluted 20x with 5% HNO_3) was repeatedly analyzed over a period of 6 hours of continuous measurement. The resulting stability plot is shown in Figure 4.1. Below shows, that excellent stability was achieved. All elements except iron (Fe) which was not found have arelative standard deviation (% RSD) of less than 0.5 % over 6 hours.

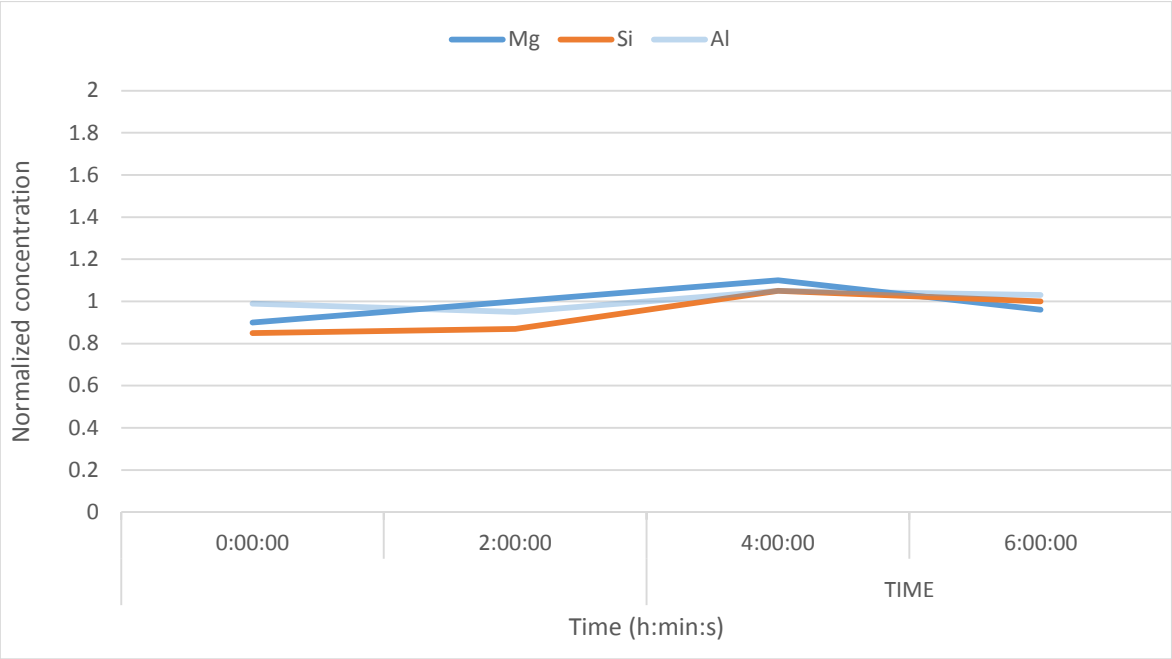

Figure 4.1 Normalized concentration of Mg, Si, and Al in a clear juice sample over 6 hours

Table 4.5 Clear juice (CJ) sample analysis result of new WSSF

S/N	Characteristics tested	Tested Method	Test result ppm(mg/L)	Standard value (mg/L)
1	Magnesium (Mg)	MP-AES	0.036 ± 0.22	3 – 24.71
2	Silicon (Si)	MP-AES	0.018 ± 0.24	78 – 315
3	Iron (Fe)	MP-AES	-	0.3 – 62
4	Aluminum (Al)	MP-AES	0.10 ± 0.36	1.5 -27
5	Total Solid	ES817:2012	19.14± 0.32	-

* mg/Kg = ppm

Table 4.6 Calcium Oxide (CaO) content of CJ sample analysis result of new WSSF of campaign 2019/2020

Month	Average ± SD CaO content in mg/L	Standard value in mg/L
November	561.4 ± 2.97^{c}	20 -150
December	616.4 ± 2.88^{a}	
January	504.4 ± 2.07^{f}	
February	567 ± 1.58^{b}	
March	556 ± 1.41^{d}	
April	535 ± 1.58^{e}	

*Mean **±** SD that do not share a letter are significantly different.

The CaO content of CJ sample is indicated in Table 4.6. The result revealed that the total CaO content is high in each month and high above the standard value (20 – 150 mg/L). Therefore, this will increase the lime salt and increase the scale formation on MEEs.Moreover, One-way ANOVA result of CaO versus months done and the P- value < 0.001 (See the appendix). Therefore, there is highly significant difference of CaO between six months. The CaO found in CJ is a prime scale former on new WSSF multiple effect evaporators. Ataklti Kahsay and Nigus Gabbiye (2015) have conducted characterization of scale formation by collected scale samples from Wonji-Shoa Sugar Factory have investigated that the deposit is clearly a scale which is formed due to Calcium compound which has higher content in the juice than iron and magnesium, in the last effect, which is critical point for heat transfer declined, in that resulted in hard scale formation.Divya Srivastava (2016) has characterized the evaporator scale of Indian sugar industry and revealed that salts of calcium are predominantly found in the first effects. Moreover, she noticed that Calcium is introduced into the juice via the cane plant and through lime addition during juice clarification. Calcium oxalate monohydrate is an inverse soluble salt and its deposition, on the hot surface of the tubes, depends upon its solubility, which in turn is a function of concentration of juice. East, Doherty, and Fellows (2015) have worked on Scale in Sugar Juice Evaporators: Types, Cases, and Prevention and investigated that Calcium phosphates deposit more heavily in the first effects due to the higher temperatures and lower sucrose concentrations. The case study of Australian cane sugar mill Doherty generalized that in any evaporator set form the first to the last vessel, the phosphates and

organic matter content of the scale decreases, while the amorphous silica and oxalate content increases. Doherty reasoned that phosphates precipitated in the earlier effects due to the slowness of reaction between lime and inorganic phosphate during clarification, causing both to carry over to the evaporators. Doherty further explained that oxalates, aconitates, silica, and silicates are formed in the late vessel due to temperature, concentration effects, and the effect of sucrose on solubility.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusion

Scale formation is of interest to the sugar industry and many areas of industry where heat transfer and flows are affected by the formation of this scale. The scale decreases the heat transfer rates in heating media and can cause severely depleted flow through piping if it forms thick scale layers in certain industries. In the sugar industry, this scale needs to be removed from heat transfer surfaces to allow the rate of heat transfer to achieve required throughputs. This cleaning means downtime for mills and costs a significant amount of money in chemicals and lost production. Since the scaling causes a reduction in heat transfer efficiency, equipment typically is oversized to account for a certain amount of scaling over time; this increases capital costs of equipment.

The study was initiated from the existing new WSSF multiple effect evaporator scale problem which is affecting the factory's performance, down time for mills, low condensate production for both sugar processing and boiler steam generation, the cost of chemicals, the cost of scrapping materials, and the cost of laborers is high.This study has been conducted by analyzing three different samples from different sources of new WSSF. These were clarified water, clarified juice, and MEEs' scale.

The clarified water which have been used for chemical (Caustic Soda, NaOH) preparation and boiling. The result showed the fluoride value (3.57 ± 0.06 mg/L) which is doubled the EU Directives surface water regulations (1989) concentration (1.7 mg/L); these is due to the nature of the area, rift valley. Moreover, the clarified water was analyzed for total hardness (TH) and the result showed that it is 39.17 ± 7.98 mg/L as compared to the standard value 0 – 300 mg/L. Hence, clarified waterhas no impact on scale formation.

The clarified juice is obtained from clarification process and is an input material for MEEs. It is characterized for Mg, Si, Fe, and Al using MP – AES test technique. Moreover, total solids, and CaO content of clarified juice was determined. The result showed that only total solid and CaO content was high. The total solid is directly related to the quantity of scale formation unless there is efficient clarification. Moreover, the one-way ANOVA result revealed that the CaO versus the months are highly significantly different ($P < 0.001$).

In WSSF's the clarification process is relatively poor as practically observed because of crushing rate was below the target due to different reasons and this will lead to liquidate the juice rather than overflow mode and this impacted to have a turbulent clear juice. Moreover, frequent failure of automatic pre-liming controller, and poor raw juice sulphitation have resulted in higher CaO content of clear juice. Therefore, the total solids % and the CaO content of clear juice resulted in scale formation on MEEs.

The scale collected from new WSSF multiple effect evaporators was analyzed for SiO_2, Fe_2O_3, CaO, MgO, Na_2O, MnO, P_2O_5, TiO_2, H_2O, and LOI and have shown thatSiO_2 content of the scale is increased from first evaporator (3.28 ± 0.014 %) to last evaporator (16.02 ± 0.007%). The Fe_2O_3 content of the scale also increases from first evaporator (1.66 ± 0.012 %) to last evaporator (38.54 ± 0.013%). CaO content of the scale is decreasing from first evaporator (25.33 ± 0.011 %) to the last evaporator (13.10 ± 0.014 %). P_2O_5 is relatively high in the first evaporator that is first evaporator (2.07 ± 0.011 %) and second evaporator (1.11 ± 0.012 %). LOI (Loss on ignition) for the samples was attributed to volatilization of organic matter, adsorbed water, water of crystallization, and loss of oxygen from the inorganic compound. As the result showed, LOI is decreasing from the first evaporator (60.70 ± 0.013 %) to the last evaporator (31.28 ± 0.03 %).

One- way ANOVA result revealed that there were very highly significant variations ($P < 0.001$) between evaporators with respect to SiO_2, Fe_2O_3, CaO, MgO, Na_2O, MnO, P_2O_5, H_2O, and LOI. However, there was no significant variation ($P > 0.05$) between evaporators with respect toTiO_2.

In general, new WSSF, multiple effect evaporators the current problem have been affected due to scale formation and heavy accumulation on the first two evaporators in particular and light scale on the last evaporators. From the analysis result, we can conclude that the source of scale is solely from the clear juice obtained from clarification process and is the source of CaO, P_2O_5, and high content of organic matters/volatile substances. Moreover, the reason for this is poor clarification practice due to intermittent raw juice sulphitation and frequent failure of automatic milk of lime dosing system. The analysis result shown the first evaporators have high content in CaO, P_2O_5, and total volatile substances. However, the scale nature of the first evaporators is theoretically soft and easily removable

by chemical or mechanical means because the phosphates and organic matter will precipitate at high temperature. Therefore, only water circulation during no or shortage of clear juice in the first evaporators makes the scale accumulation high and even harder as compared to the previous old Wonji/ Shoa sugar factories. The last evaporators as the result exposed high content in SiO_2 and sesquioxides, like Fe_2O_3. Even though the percentage of SiO_2 content is high in scale composition, it is the nature of these oxides that will precipitate out with concentrated juice/syrup at last evaporators. Since the amount of deposition of these impurities is small, no scale removal problem faced so far.

5.2. Recommendations

- For new WSSF frequent failure of automatic pre-liming controller, proper sulphitation, and avoiding juice liquidation while crushing is going on in order to have an efficient clarification system because it is very crucial to remove maximum possible impurities like suspended and soluble solids in the juice and indirectly it will reduce the severity of scale formation on MEEs.

- From the laboratory result, the first evaporators' scale is seen to have high amount of CaO, P_2O_5, and LOI. This indicates that the scale is with high content of phosphate and organic matters. So, for this the most recommended caustic soda solution of 25 – 30 ^{O}Bx, containing 10 % Na_2CO_3 and 5 – 10 % NaCl the effect of chemical cleaning will be more pronounced instead of using random concentration of caustic soda and neglecting use of soda ash and common salt.

- To avoid heavy scale accumulation on the first two evaporators(Evap#1 and Evap#2)of either set of evaporator water circulation during no clear juice or due to any other reason it is a very good practice to circulate in all evaporators of the set because the recirculation on the first evaporators having high temperature will rapidly concentrate during high retention time.

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7. APPENDIXES

Appendix Table 1 Thermal Conductivity (λ) in W/m K

Copper	393
Aluminum (Pure)	221
Duralumin	146
Brass	90 – 110
Iron (Pure)	67
Gray cast iron	42 – 63
Steel 0.2 % C	50
Steel 0.6 % C	46
Stainless steels 304,316, and 321	15
Inorganic scale	0.08 – 2.20
Organic film	0.41 – 0.95
Oil deposits	0.12 – 0.29

Appendix Table 2 Effect of temperature and sucrose concentration on solubility

Scaling Phase	**Increasing temperature**	**Increasing sucrose concentration**
Silica	Increased solubility	Decreased solubility
Calcium Oxalate	Increased solubility	Decreased solubility
Calcium sulphate dihydrate	Decreased solubility	Decreased solubility
Phosphates (Ca)	Decreased solubility	Increased solubility
Calcium Carbonate	Decreased solubility	Increased solubility
Aconitates (Ca, Mg)	Increased solubility	Decreased solubility

Appendix Table 3 New Wonji/Shoa sugar factory clarified water and clarified juice sample analysis result

CLARIFIED WATER ANALYSIS RESULT								
TESTS							**AVERAGE**	**STDEV**
Sodium	75	66	73				71.33	4.72
Potassium	18	12	18				16	3.46
Total iron	0.67	0.83	0.55				0.68	0.14
Chloride	36.08	38.27	40.46				38.27	2.19
Sulphate	28.8	27.66	31.56				29.34	2.00
Fluoride	3.6	3.5	3.6				3.57	0.06
Phosphate	0.14	0.18	0.16				0.16	0.02
Total Hardness	30	45	32	50	43	35	39.17	7.98
CLARIFIED JUICE CaO CONTENT RESULT								
MONTHS								
November	562	560	566	558	561		561.4	2.97
December	620	617	616	617	612		616.4	2.88
January	502	506	503	504	507		504.4	2.07
February	566	567	568	565	569		567	1.58
March	555	557	556	558	554		556	1.58
April	535	534	533	536	537		535	1.58

Cont'd

CLEAR JUICE ANALYSIS RESULT				
Clear juice Long Term Stability				
TIME	**Mg**	**Si**	**Fe**	**Al**
0:00:00	0.9	0.85	-	0.99
2:00:00	1.0	0.87	-	0.95
4:00:00	1.1	1.05	-	1.05
6:00:00	0.96	1.0	-	1.03
Element concentration (mg/L)				
	0.03	0.02	-	0.15
	0.047	0.012	-	0.08
	0.031	0.022	-	0.07
AVERAGE	0.036	0.018	-	0.1
STDEV	0.007789	0.0043205	-	0.03559
% RSD	0.22	0.24	-	0.36

Appendix Figure 1 New WSSF heating calandria tubes manual cleaning system

Appendix Figure 2 Collected scale samples from new WSSF multiple effect evaporators

One-way ANOVA: SiO_2 versus EVAPORATOR

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05
Rows unused	2

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
EVAPORATOR	4	E1, E2, E3, E5

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	3	253.210	84.4033	519405.00	0.000
Error	4	0.001	0.0002		
Total	7	253.211			

Model Summary

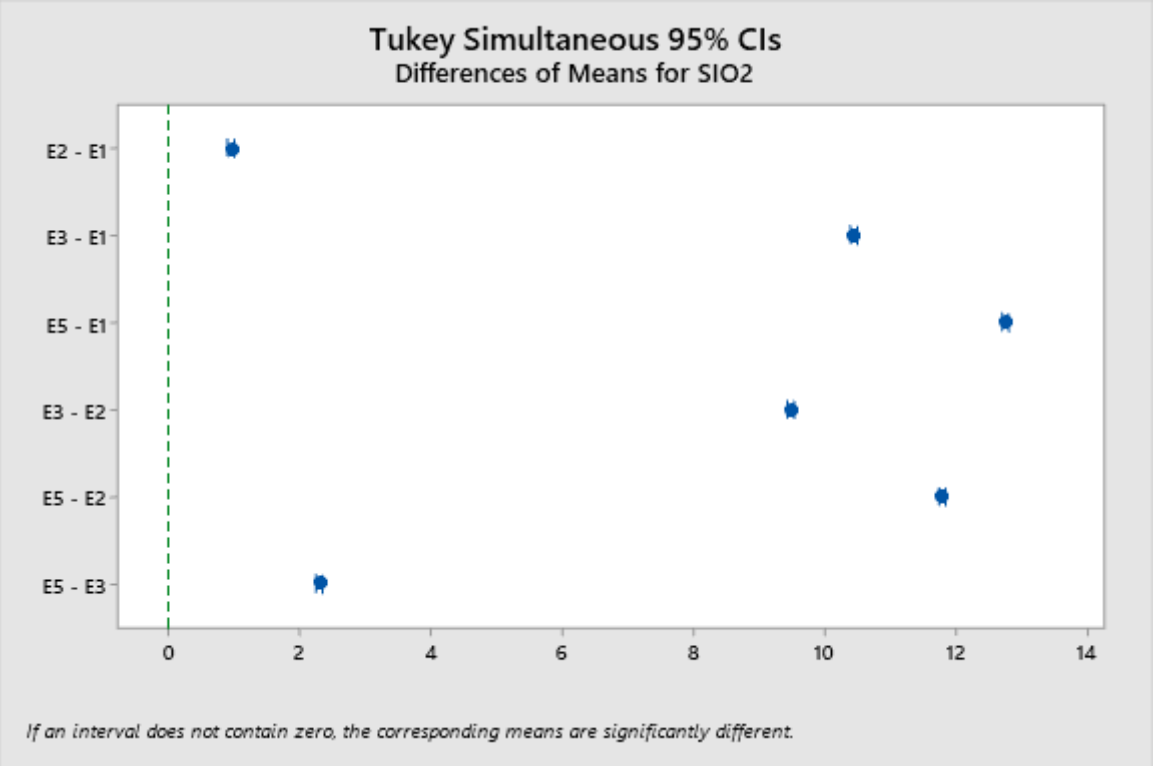
S	R-sq	R-sq(adj)	R-sq(pred)
0.0127475	100.00%	100.00%	100.00%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E5	2	16.0250	A
E3	2	13.7200	B
E2	2	4.2400	C
E1	2	3.2800	D

Means that do not share a letter are significantly different



One-way ANOVA: Fe_2O_3 versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	1520.27	380.067	1900337.20	0.000
Error	5	0.00	0.000		
Total	9	1520.27			

Model Summary

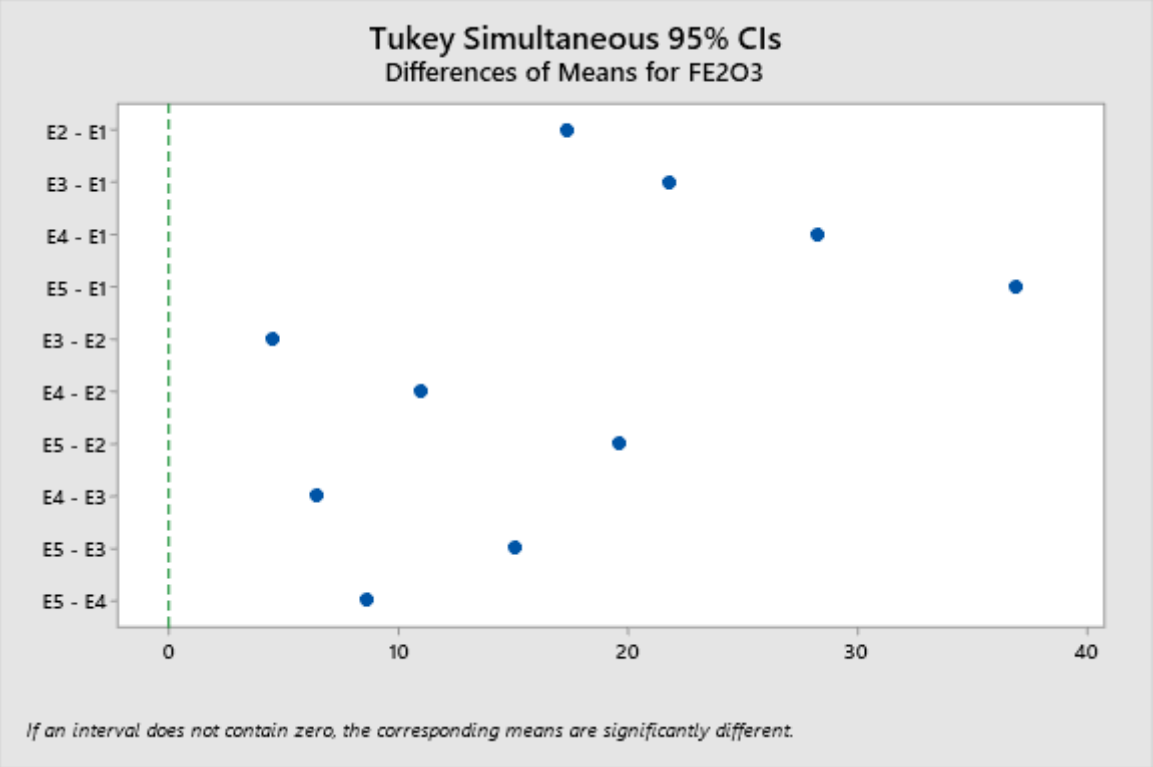
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	100.00%	100.00%	100.00%

.Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E5	2	38.5400	A
E4	2	29.9200	B
E3	2	23.4800	C
E2	2	18.9600	D
E1	2	1.6600	E

Means that do not share a letter are significantly different.



One-way ANOVA: CaO versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	259.870	64.9676	324837.80	0.000
Error	5	0.001	0.0002		
Total	9	259.871			

Model Summary

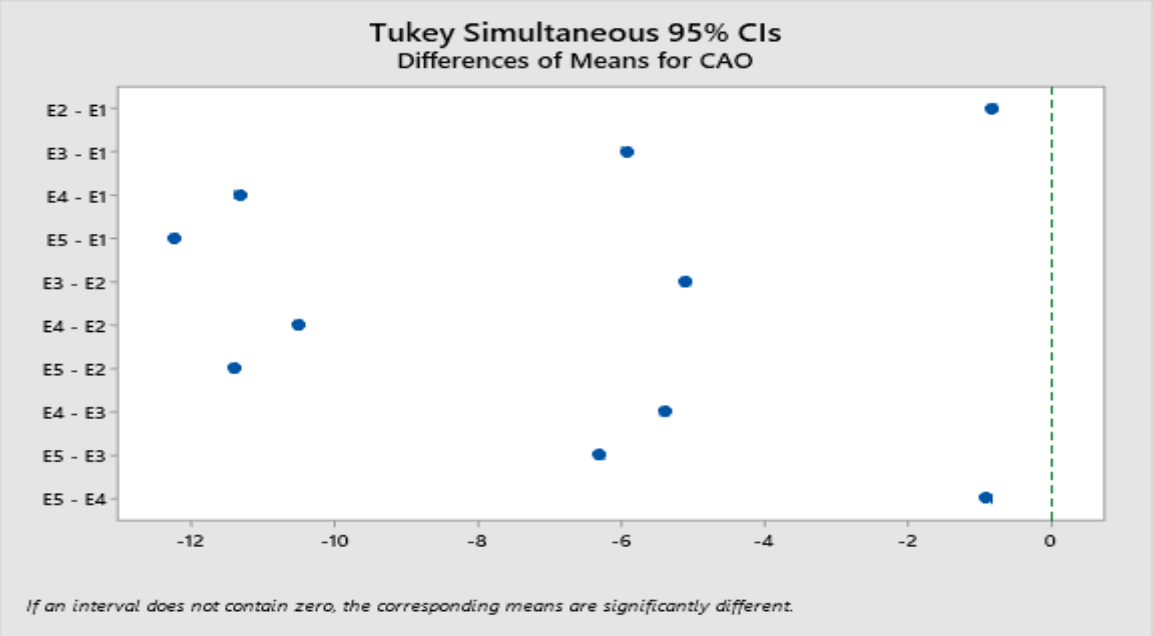
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	100.00%	100.00%	100.00%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E1	2	25.3300	A
E2	2	24.5000	B
E3	2	19.4000	C
E4	2	14.0000	D
E5	2	13.1000	E

Means that do not share a letter are significantly different.



One-way ANOVA: MgO versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	0.691200	0.172800	864.00	0.000
Error	5	0.001000	0.000200		
Total	9	0.692200			

Model Summary

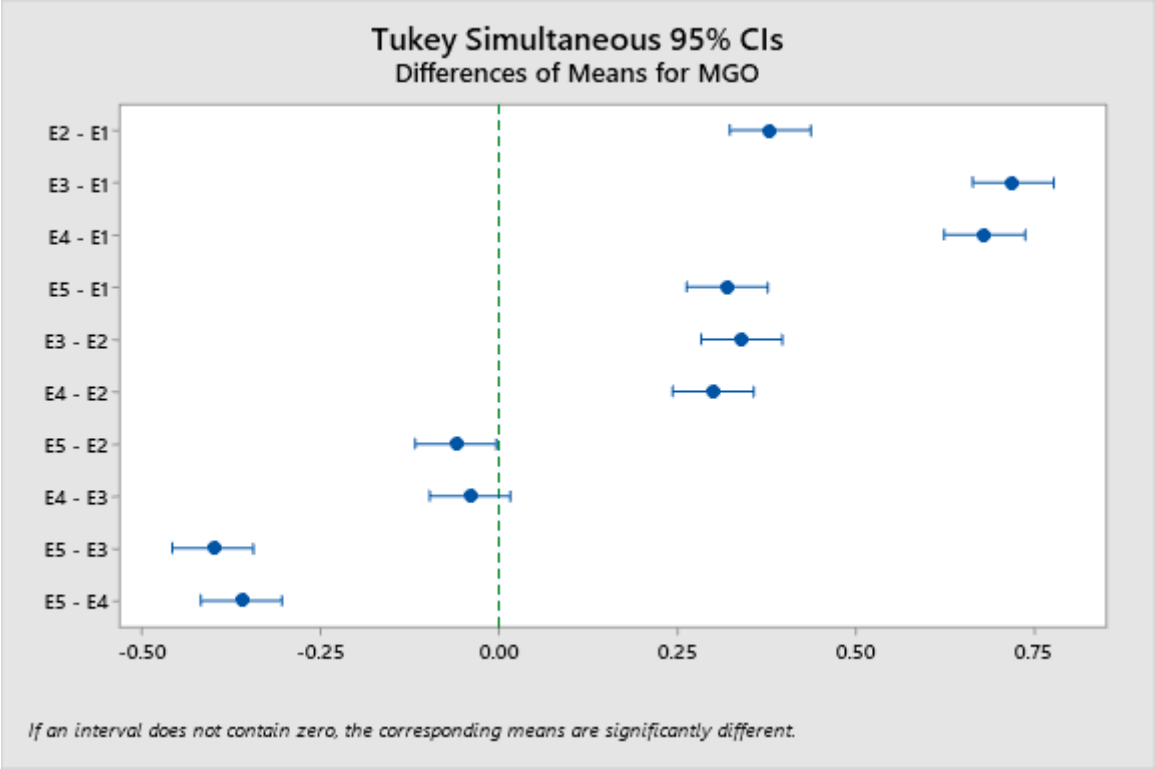
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	99.86%	99.74%	99.42%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping		
E3	2	0.8200	A		
E4	2	0.7800	A		
E2	2	0.4800		B	
E5	2	0.4200			C
E1	2	0.1000			D

Means that do not share a letter are significantly different.



One-way ANOVA: Na_2O versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	6.14944	1.53736	7686.80	0.000
Error	5	0.00100	0.00020		
Total	9	6.15044			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	99.98%	99.97%	99.93%

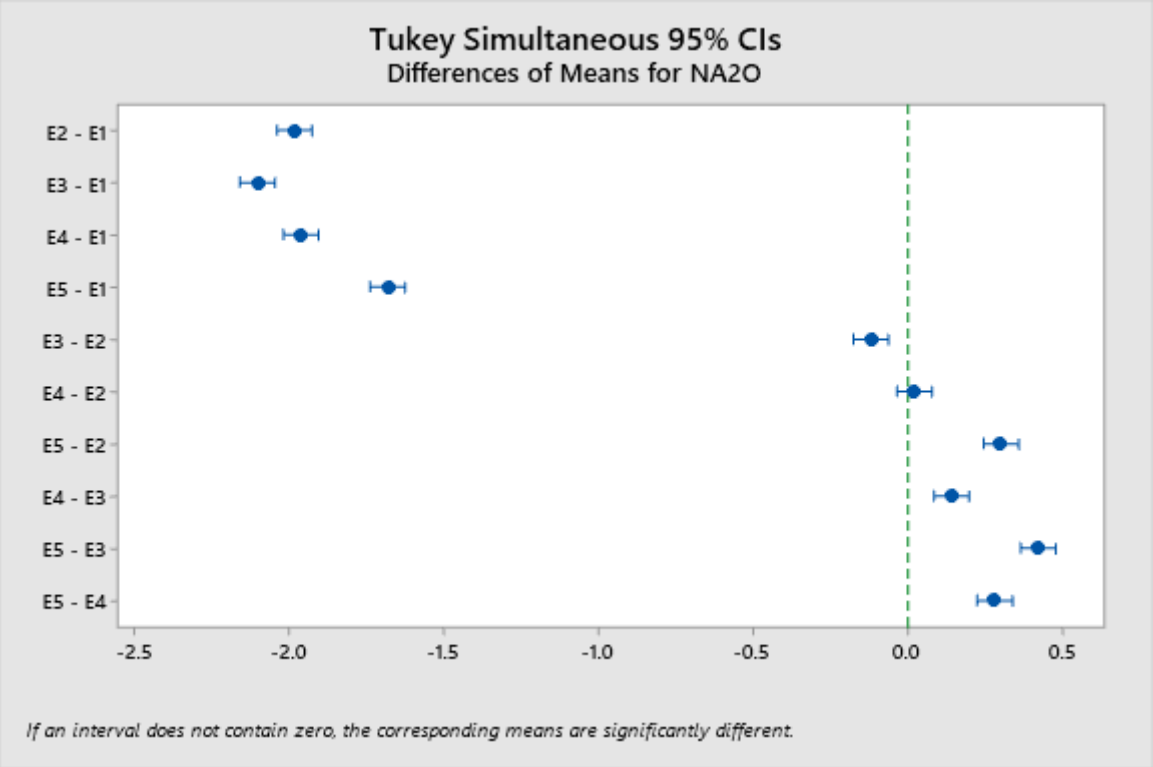
Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping		
E1	2	2.1600	A		
E5	2	0.4800		B	
E4	2	0.2000			C

E2	2	0.1800	C	
E3	2	0.0600		D

Means that do not share a letter are significantly different.



One-way ANOVA: MnO versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	0.096960	0.024240	121.20	0.000
Error	5	0.001000	0.000200		
Total	9	0.097960			

Model Summary

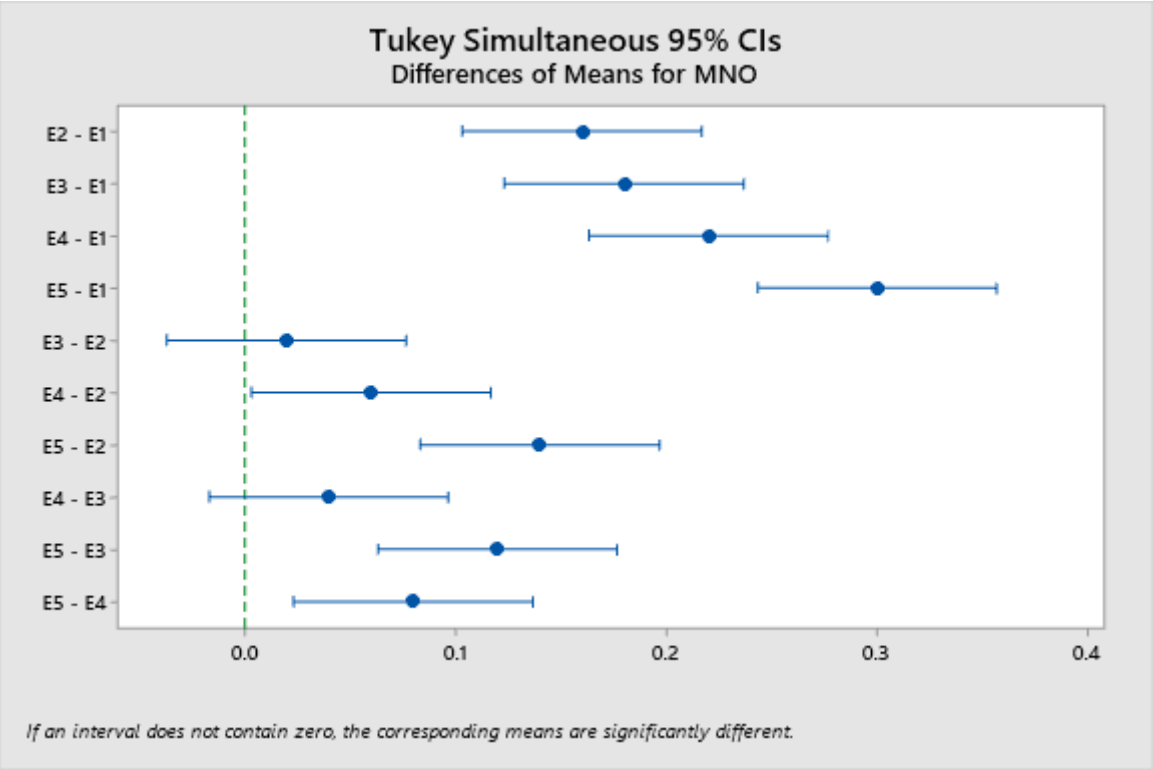
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	98.98%	98.16%	95.92%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping		
E5	2	0.4400	A		
E4	2	0.3600		B	
E3	2	0.3200		B	C
E2	2	0.3000			C
E1	2	0.1400			D

Means that do not share a letter are significantly different.



One-way ANOVA: P_2O_5 versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	3.38440	0.846100	4230.50	0.000
Error	5	0.00100	0.000200		
Total	9	3.38540			

Model Summary

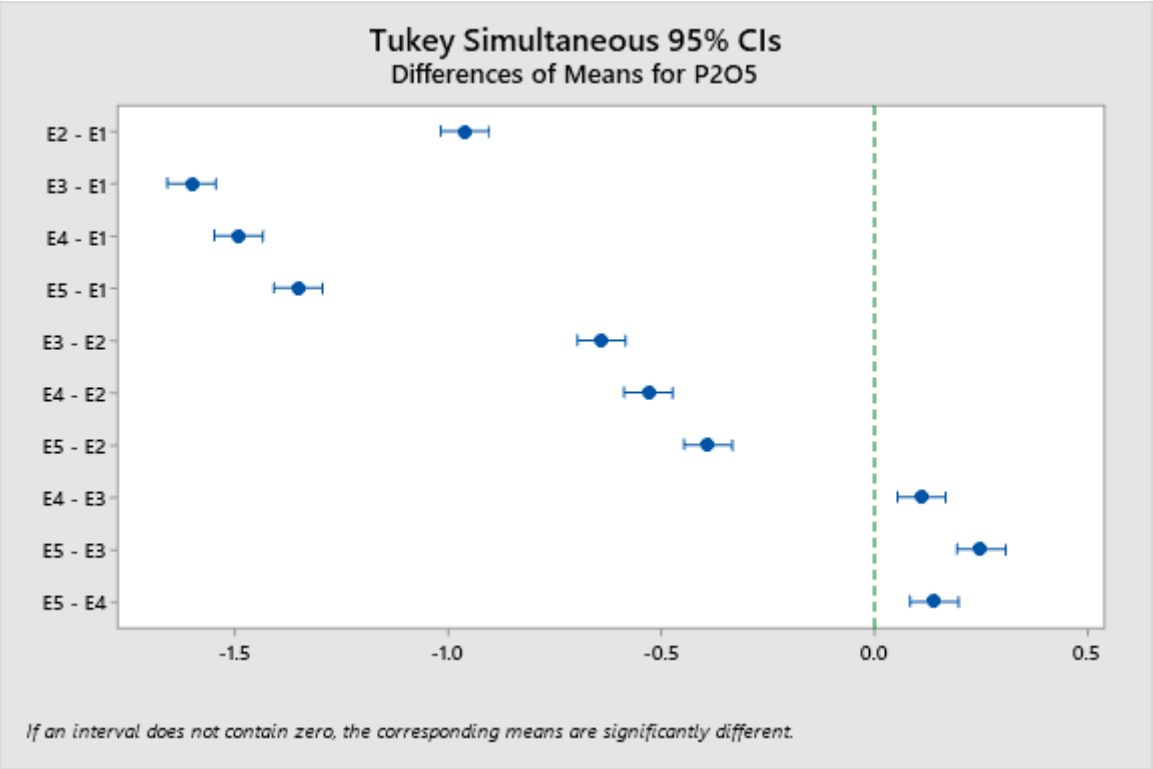
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	99.97%	99.95%	99.88%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E1	2	2.0700	A
E2	2	1.1100	B
E5	2	0.7200	C
E4	2	0.5800	D
E3	2	0.4700	E

Means that do not share a letter are significantly different.



One-way ANOVA: TiO_2 versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	0.001360	0.000340	1.70	0.285
Error	5	0.001000	0.000200		
Total	9	0.002360			

Model Summary

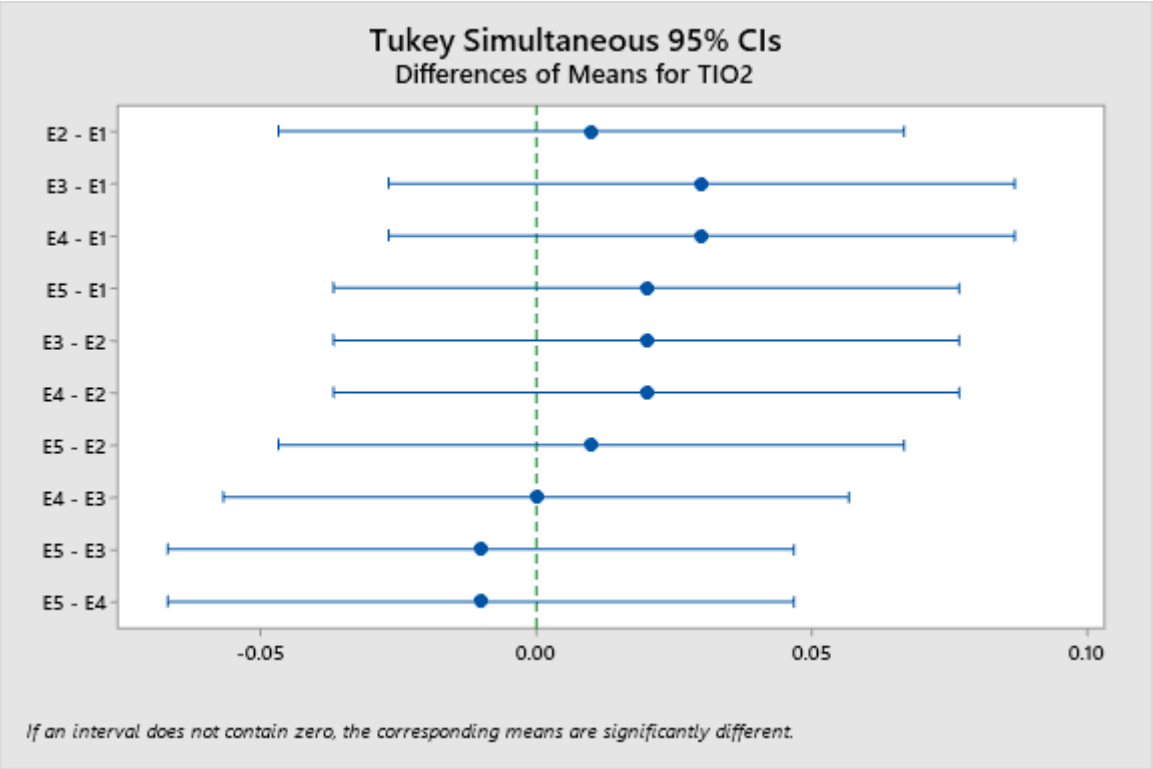
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	57.63%	23.73%	0.00%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E4	2	0.0400	A
E3	2	0.0400	A
E5	2	0.0300	A
E2	2	0.0200	A
E1	2	0.0100	A

Means that do not share a letter are significantly different.



One-way ANOVA: H_2O versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	5.10136	1.27534	6376.70	0.000
Error	5	0.00100	0.00020		
Total	9	5.10236			

Model Summary

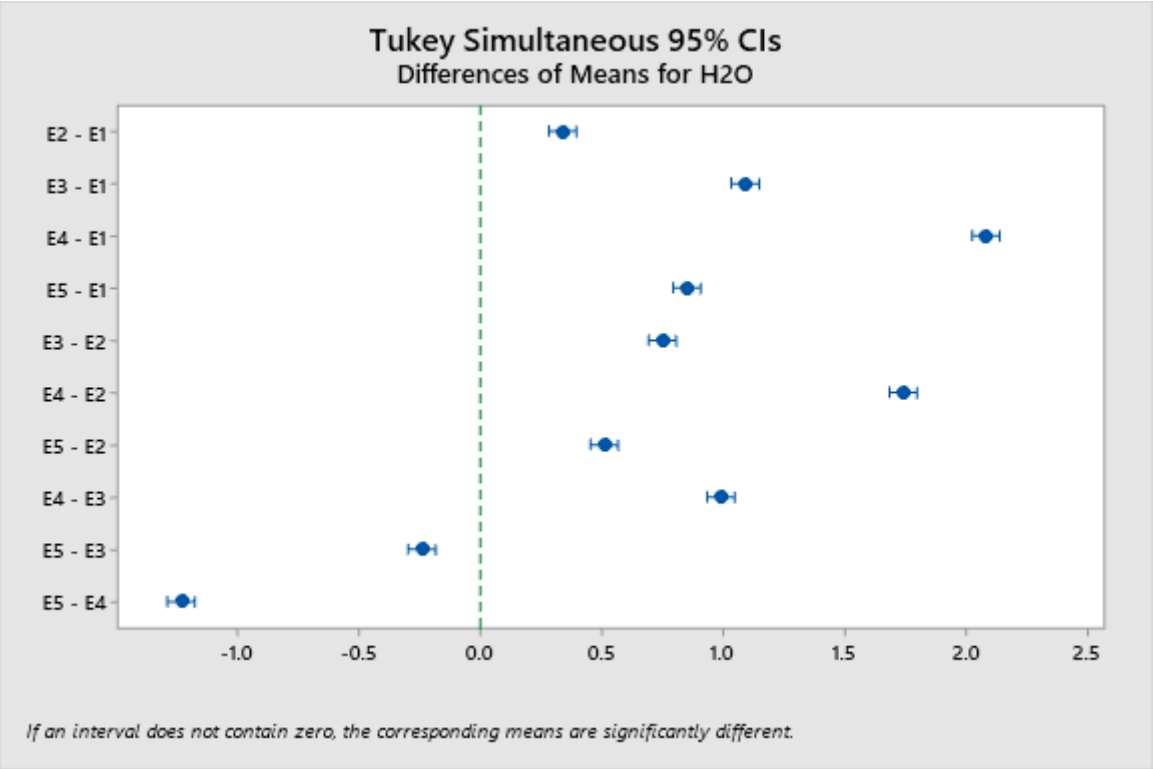
S	R-sq	R-sq(adj)	R-sq(pred)
0.0141421	99.98%	99.96%	99.92%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E4	2	4.6200	A
E3	2	3.6300	B
E5	2	3.3900	C
E2	2	2.8800	D
E1	2	2.5400	E

Means that do not share a letter are significantly different.



One-way ANOVA: LOI versus EVAPORATOR

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
EVAPORATOR	4	1163.49	290.872	1163486.64	0.000
Error	5	0.00	0.000		
Total	9	1163.49			

Model Summary

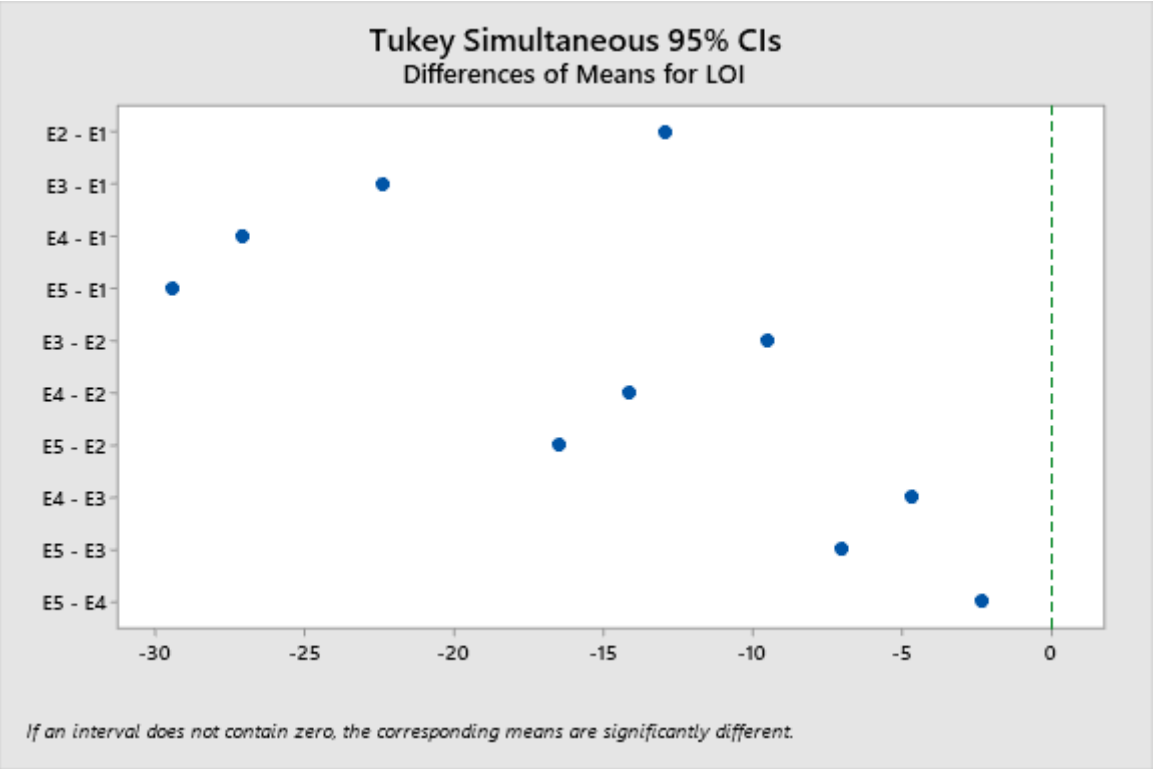
S	R-sq	R-sq(adj)	R-sq(pred)
0.0158114	100.00%	100.00%	100.00%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

EVAPORATOR	N	Mean	Grouping
E1	2	60.7000	A
E2	2	47.7800	B
E3	2	38.2750	C
E4	2	33.6100	D
E5	2	31.2800	E

Means that do not share a letter are significantly different.



One-way ANOVA: CaO versus Month

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
MONTH	5	34494.7	6898.94	1432.31	0.000
Error	24	115.6	4.82		
Total	29	34610.3			

Model Summary

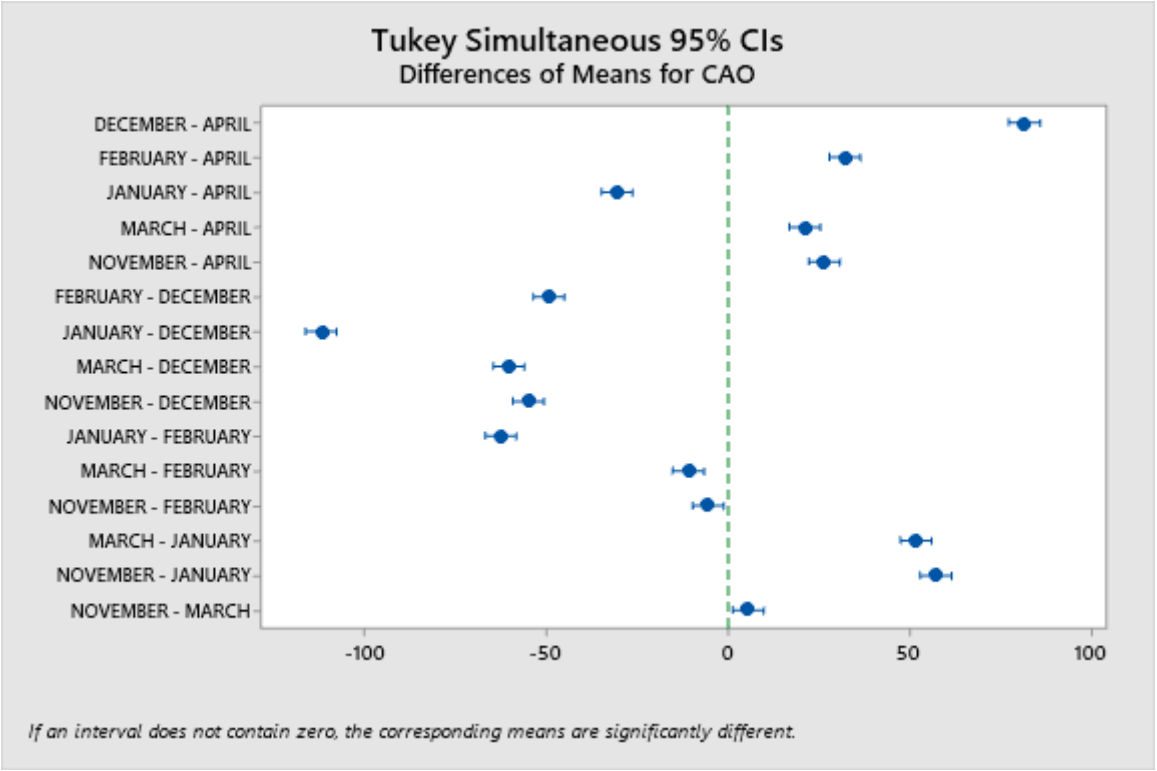
S	R-sq	R-sq(adj)	R-sq(pred)
2.19469	99.67%	99.60%	99.48%

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

MONTH	N	Mean	Grouping
DECEMBER	5	616.40	A
FEBRUARY	5	567.000	B
NOVEMBER	5	561.40	C
MARCH	5	556.000	D
APRIL	5	535.000	E
JANUARY	5	504.400	F

Means that do not share a letter are significantly different.


Tukey Simultaneous 95% CIs
Differences of Means for CAO
DECEMBER - APRIL
FEBRUARY - APRIL
JANUARY - APRIL
MARCH - APRIL
NOVEMBER - APRIL
FEBRUARY - DECEMBER
JANUARY - DECEMBER
MARCH - DECEMBER
NOVEMBER - DECEMBER
JANUARY - FEBRUARY
MARCH - FEBRUARY
NOVEMBER - FEBRUARY
MARCH - JANUARY
NOVEMBER - JANUARY
NOVEMBER - MARCH
-100
-50
0
50
100
If an interval does not contain zero, the corresponding means are significantly different.