

 Sudan University of Science and Technology

College of Graduate Studies and Scientific Research

Manufacture of basic chromium sulphate and its impact on the environment

تصنيع كبريتبت الكروم القبعدية وأثرهب علي البيئة

A thesis submitted in partial fulfillment of the requirements for The M.Sc. Degree in chemical engineering

BY

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بسم الله الرحمن الرحيم

قال تعالى:

(وَإِرْ قَالَ رَبُّكَ لِلمَلائِكَةِ إِنِّي جَاعِلٌ فِي الأَرْضِ خَلِيفَةً قَالُوا أَتَجْعَلُ فِيهَا مَنْ يُفْسِدُ فِيهَا وَيَسْفِكُ الدِّمَاءَ وَنَحْنُ نُّسَبِّحُ بِحَمْدِكَ وَنُقَدِّسُ لَكَ قَالَ إِنِّي أَعْلَمُ مَا لا تَعْلَمُونَ (30) وَعَلَّمَ آدَمَ الأَسْمَاءَ كُلَّهَا ثُمَّ عَرَضَهُمْ عَلَى الْمَلائِكَةِ فَقَالَ أَنْبِئُونِي بِأَسْمَاءِ هَؤُلاء إِنْ كُنْتُمْ صَادِقِينَ(31) قَالُوا سُبْحَانَكَ لا عِلْمَ لَنَا إِلاَّ مَا عَلَمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ(32))

صدق الله العظيم

اآليبث)03-03 (سىرة اىبقرة

Dedication

To my mother

 To my father

 To my brothers and sisters

To all my family

To all my teachers

 To all my colleagues and friends

With love and respect.

YOUSIF

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 All thanks are due to Almighty Allah (SWT) who gave me health and strength, and helped me tremendously to produce this work.

 I would like to express my thanks to my supervisor prof: Hajo College of engineering, Sudan University of science and technology, who devoted most of his time to teach us on various disciplines of scientific research. Also, I offer my thanks to the Department of Chemical Engineering and all the professors.

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ABSTRACT

 In this study, basic chromium sulfate was prepared, and the product was manufactured from the widely used triple chromium, in addition to sodium chromate and calcium chromate in certain proportions at a temperature of 1200 °C, as well as using sulfuric acid and glucose as shown in the experiment. Manufacturing until we get the final product. Some of the devices used in the mass production process, such as the CSTR reactor and dryer, were designed, with a brief explanation of the rest of the devices used. In addition, a study was conducted on the environmental impact of the product, in proportion to the industrial need and to achieve the slogan of clean and environmentally friendly work. Although there is no environmental impact of the product that has been manufactured, care must be taken when dealing with these materials as well as safety and health precautions must be used professional establishments.

 A sample of the skin was taken and treated with the basic chromium sulfate that was manufactured, and some chemical tests were conducted on the sample, and the conclusion is that the base percentage is 33.33%, the percentage of chromium oxide is 3.8%, and the total ash percentage is 2.25%, and these percentages are evidence of the quality of the product.

المستخلص

في هذه الدراسة ، تم تحضير كبريتات الكروم الأساسية ، وتم تصنيع المنتج من الكروم الثلاثي المستخدم على نطاق واسع ، بالاضافة لكرومات الصوديوم وكرومات الكالسيوم بنسب معينة عند درجة حرار 0° 1200 درجة مئوية ، وكذلك استخدم ح*مض* الكبريتيك والجلكوز حسب ماهو موضح في التجربة ، وتم ضبط خطوات التصنيع حتى تحصلنا على المنتج النهائي . وتم تصميم بعض الأجهزة المستخدمة في عملية الإنتاج بالجملة ، مثل مفاعل CSTR والمجفف ، مع شرح موجز لبقية للأجهزة المستخدمة ، وتم عمل مخطط يجمع كل هذه الاجهزة بالترتيب بواسطة برنامج الهايسس ، للاستفادة منه عند تحويل المشروع لمصنع كبريتات الكروم القاعدية . بالإضافة إلى ذلك ، أجريت دراسة حول التأثير البيئي للمنتج ، وذلك نسبة للحوجة الصناعية ولتحقيق شعار العمل النظيف والصديق للبيئة على الرغم من عدم وجود اوقلة التأثير البيئي للمنتج الذي تم تصنعه ، يجب توخَّى الحذر عند التعامل مع هذه المواد وكذلك لابد من استخدام ً احتياطات السلامة والصحة المهنية التي تضعها المؤسسات .

تم أخذ عينة من الجلد ومعالجتها بواسطة كبريتات الكروم القاعدية التي تم تصنيعها ، كما تم إجراء بعض الإختبارات الكيميائية على العينة ، وخلاصة ذلك ان نسبة القاعدية % 33.33 ، ونسبة أوكسيد الكروم 3.8% ، ونسبة الرماد الكلِّي % 2.25 ، وهذه النسب دليل على جودة المنتج .

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All symbols are defined in the text when they are first used

 $CF = specific heat$ o

 $F=$ Feed, kcal/kg \circ C

 CP_1 , CP_2 , CP_3 , = specific heat of Product in effects 1 to 3, kcal/kg ∘C

 C_{C1} , C_{C2} , C_{C3} , = specific heat of Condensate in effects 1 to 3, kcal/kg .◦C

 λ 0 = Latent heat of Steam (to 1st effect), kcal/kg

 $\lambda_1, \lambda_2, \lambda_3$, = Latent heat of water evaporated, Kcal/kg

 $TF = Temperature of Feed, °C$

 T_0 = saturation temperature of feed to first effect, ⁰C

 T_1, T_2, T_3 , = Temperature at which evaporation takes place in effects 1 to 3, kJ/kg ∘C

 U_1 , U_2 , U_3 =Over all heat transfer coefficient KW/m₂.K.

 T_{P1} , T_{P2} , T_{P3} , = Product outlet temperature in effects 1 to 3, kJ/kg ∘C

 $MF = Mass$ flow rate of feed, kg/S

 $Xf =$ Initial Total Dissolved Solids

 xp_1, xp_2, xp_3 Dissolved Solids in effects 1 to 3

 $MP = Mass$ flow rate Product should be, kg/S

 $ME = Total water evaporated, kg/S$

 $SE = Steam Economy$

 D_0 = Mass flow rate of steam, kg/S

 D_1 , D_2 , D_3 = water removed in effects 1 to 3, kg/S

 m_{P1} , m_{P2} , m_{P3} , $m_{P4} =$ Mass flow rate of Product obtained in effects 1 to 3, kg/S

 m_{C1}, m_{C2}, m_{C3} , = Mass flow rate of condensate obtained in effects 1to 3, Kg/S

 $O.D =$ outside of diameter

NT =No of tubes

The design equation for CSTR is:-

 FA_{0} = feed flow rate (kg/h)

 $FA = product flow rate (kg/h)$

 $X =$ conversion

 $-r_A$ = rate of the reactor

 $V =$ volume of CSTR

Where

t=minimum designed thickness (mm)

P= internal pressure (N/mm^2)

Di = vessel internal diameter (mm)

F= the design stress

Take thickness = 10 mm for stainless steel

CHAPTER ONE

1. Introduction

 [Chromium](http://www.chemistryexplained.com/knowledge/Chromium.html) was first identified in 1797 by the French chemist Louis-Nicolas, a mineral also called Siberian red lead. The name for chromium is taken from the Greek chromo, which means "color." This is a fitting name, because chromium compounds are often found in vividly colorful shades of green, red, or yellow. As such, chromium compounds historically have found extensive use as pigments for paints. In contrast, [elemental](http://www.chemistryexplained.com/knowledge/Elemental.html) chromium is a shiny, hard, yet brittle, steel-gray metal. Since chromium is not found naturally in its free elemental state, it is usually extracted from chromites, Fe Cr $2O_4$. Chromium is also an important alloy component in stainless steel because it makes the steel stronger and more corrosion-resistant. Paint pigments account for more than one-third of all chromium usage each year. Overall, chromium is the twentyfirst most abundant element in Earth's crust. It is usually found in the +2 (e.g., Cr_2O_3), +3 (e.g., Cr_2O_3), or +6 (e.g., $K_2Cr_2O_7$). [\(Krebs, 2006\)](#page-64-0)

1.1 General description

1.1.1 Sources

 Chromium (Cr) is a grey, hard metal most commonly found in the trivalent state in nature. Hexavalent (chromium (VI)) compounds are also found in small quantities. Chromite (FeOCr₂O₃) is the only ore containing a significant amount of chromium. The ore has not been found in the pure form; its highest grade contains about 55% chromic oxide[\(IARC, 1980\)](#page-64-1) . In Europe, ferrochromium is produced mainly in Finland, France, Italy, Norway, Sweden and former Yugoslavia. Potassium chromate is produced mainly in Germany, Italy, Switzerland and the United Kingdom. Sodium chromate and dichromate are now among the most important chromium products, and are used chiefly for manufacturing chromic acid, chromium pigments, in leather tanning and for corrosion control[\(IARC, 1990\)](#page-64-2),[\(LANGÅRD, 1990\)](#page-64-3).Chromium levels in soil vary according to area and the degree of contamination from anthropogenic chromium sources. Tests on soils have shown chromium concentrations ranging from 1 to 1000 mg/kg, with an average concentration ranging from 14 to about 70 mg/kg [\(Park, 1984\)](#page-64-4) . Chromium (VI) in soil can be rapidly reduced to chromium (III) by organic matter. As chromium is almost ubiquitous in nature, chromium in the air may originate from wind erosion of shale, clay and many other kinds of soil. In countries where chromite is mined, production processes may constitute a major source of airborne chromium. In Europe, endpoint production of chromium compounds is probably the most important source of chromium in air.

1.1.2 Occurrences, Uses, and Properties of Chromium

Chromium is the $21st$ most abundant element in the Earth's crust with. Although it exists in several oxidation states, the zero, trivalent, and hexavalent states are the most important in commercial products and the environment. Nearly all naturally occurring chromium is in the trivalent state, usually in combination with iron or other metal oxides. Although only about 15% of the chromium mined is used in the manufacture of chemicals, most applications of chromium utilize the chemistry of chromium. For instance, the "stainless" nature of stainless steel is due to the chemical properties of the chromium oxides which form on the surface of the alloy. Similarly, the protective properties of chrome plating of metals, Chromate Copper Arsenate (CCA) treatment of wood, and chrome tanning of leather are all dependent on chromium chemistry. The key to these uses is that under typical environmental and biological conditions of pH and oxidation– reduction potential, the most stable form of chromium is the trivalent oxide. This form has very low solubility and low reactivity resulting in low mobility in the environment and low toxicity in living organisms. [\(Barnhart, 1997\)](#page-64-5)

1.1.3 Production of Chromium

Depending on the type of product being processed, chromium ore can be classified in three categories, each being used for different purposes:

1-Ores used in metallurgy

2-Ores used in production of non-melting substances and anti-corrosive substances,

3-Ores intended for use in chemistry industry.

In order for an ore intended for metallurgy to be suitable to obtain pure chromium-iron alloy, it should contain minimum 48% Chrome oxide (Cr_2O_3) , and should contain $1/3$ chromium for iron. Chrome must be degraded in order to prepare chrome alloys.

For instance, oxygen should be combined with other elements such as carbon (coke) and destroyed at mine melting pits. Alloy named Ferro-chromium which is an iron and chromium alloy is obtained in this manner. This alloy also contains some carbon. Although chromium is present in various minerals, the only resource to obtain chromium is chromites thanks to economical processing properties.

1.1.4 Colors

These compounds have values of 2 3 5 6 7. Chromium 2 compounds are blue, chromium 3 compounds are green, chromium 5 is colorless and chromium 6 and 7 compounds are yellow, orange and red. Chromium compounds are widely used in paint industry due to such qualities .[\(A1.2019\)](#page-63-0)

1.2 Basic Chromium Sulphate (BCS)

Dark green powder with high tanning properties, it dissolves easily in water and mainly used in the tanning of various leathers and furs.

1.2.1 Description of BCS

Other name: Basic chromic sulfate; chromium (iii) sulphate basic

Appearance: dark green powder

Molecular formula: $Cr \cdot (OH)_{m} \cdot (SO_4)$ n• $X (H_2O)$

Molecular weight: 343.21

1.3 Tanning processes:-

Pretannage operations of liming and deliming leave the pelt collagen at a pH 5-6, with basic chromium sulphates, reaction with the pelt would be very rapid and lead to over tanning of outermost surface unless special precaution is taken. One approach is to use masked tanning salt. Another approach is to discharge the carboxyl groups of pelt collagen by back titration with strong acid. Unionized carboxyl groups are inactive in forming Complex with the Cr (tanning action completely prevented) and hence penetration of the pelt by the chrome liquor may be achieved. The subsequent addition of alkali or highly basic Cr salt raises the pH value and tannage takes place. Excessive swelling of the pelt by acid is prevented by adding neutral salt to the pickle liquor (Balanced conditions require skill).

Particle size of Cr complexes is also of importance. It is thought that polynuclear complexes of 2 to 7 Cr atoms are present in solutions of chromium sulphate of Basicity 33- 50. It was found that at 40 % overall Basicity the addition of carboxylic acid masking agents could increase the particle size twofold. At higher Basicity, insoluble masked complexes and aggregation takes place.

 The chromium sulphates used in leather industry are predominantly cationic at concentrations normally (2 % Cr_2O_3 solutions) used. Concentrated stock liquors (11-15 % Cr_2O_3) and dry powders (25-33 % Cr_2O_3) when freshly diluted may be non-ionic or even anionic in character. Conversion to cationic character is always favored by aging of dilute solutions.

1.4 Temperature:

The control of the temperature in chrome tannage process is important, With increased temperature (up to 40°C), there is increasing in the fixation of chrome.

The temperature control in drum tannage can be best accomplished by control of the temperature of the solution entering the drum and careful attention [\(SHARAF, 2013\)](#page-64-6)

1.5 Effect of pH value:

The tanning behavior of chrome is related directly to its pH value. salt content and acidity .Basic chromium salts ,as chrome tanning , when dissolved in water , result in pH of solution of about 2.5-3.5 depending on the Basicity , concentration and nature of the salt used [\(SHARAF, 2013\)](#page-64-6)

1.6 Research problem

1. The basic chromium sulphate is imported at very high prices for tannage need hard currencies

2 .What is happening now is size reduction then exporting of raw chromium , so we can easily start manufacturing basic chromium sulphate for Sudanese tanneries and then export for those who need.

1.7 Research goals

1. To starty manufacturing basic chromium sulphate.

2 - Manufacturing of basic chromium sulphate in the laboratory can help in making feasibility study.

3 - Know the effect of pollution resulting from the use of basic chromium sulphate during the manufacturing and use and after its disposal in the surrounding environment after use.

4. Conducting a preliminary study to clarify the designs of the devices used to manufacture production quantities

1.8 Research importance

1 - The country tanneries are in need of basic chromium sulphate, which is essential in the leather industry

2 - Abundance of chromium ore in Sudan as well as the abundance of companies that exports crude chromium to convince these to manufacture chromium sulphate commercially.

CHAPTER TWO

LITERATURE REVIEW

Section One

Stages Manufacturing of Basic Chromium Sulphate

Figure (2.1) Production scheme for chromium sulphate.

2.1 Introduction

 Chromium (III) Sulfate is a moderately water and acid soluble Chromium source for uses compatible with sulfates. Sulfates are salts or esters of sulfuric acid formed by replacing one or both of the hydrogen with a [metal.](https://www.americanelements.com/metals.html) Most metal sulfate compounds are readily soluble in water for uses such as [water treatment,](https://www.americanelements.com/water-treatment-chemicals.html) unlike [fluorides](https://www.americanelements.com/Fluorides.html) and [oxides](https://www.americanelements.com/oxides.html) which tend to be insoluble. [Organ metallic](https://www.americanelements.com/organometallics.html) forms are soluble in organic solutions and sometimes in both aqueous and organic solutions.

 Metallic ions can also be dispersed utilizing suspended or coated [nanoparticles](https://www.americanelements.com/nanomaterials-nanoparticles-nanotechnology.html) and deposited utilizing [sputtering targets](https://www.americanelements.com/sputtering-targets.html) and [evaporation materials](https://www.americanelements.com/thin-film-evaporation-materials.html) for uses such as [solar](https://www.americanelements.com/solar-energy-materials.html) [cells](https://www.americanelements.com/solar-energy-materials.html) and [fuel cells.](https://www.americanelements.com/fuel-cell-materials.html) Chromium Sulfate is generally immediately available in most volumes. High purity, submicron and nanopowder forms may be considered .Chromium (III) sulfate is the inorganic compound with chemical formula $Cr_2(SO₄)₃$.[\(B1.2019\)](#page-63-1)

2.2 Chemical Properties

 Solutions of chromium (III) sulfate turn from blue to green upon heating, indicating the formation of a less reactive "sulfate-complex" that may take days or weeks to revert. Freshly prepared chromium sulfate often contains traces of this coloration, with some samples even appearing green rather than blue, but is otherwise usable.

A solution of both chromium (III) sulfate and [potassium sulfate](http://www.sciencemadness.org/smwiki/index.php/Potassium_sulfate) will yield crystals of [chrome alum](http://www.sciencemadness.org/smwiki/index.php?title=Chrome_alum&action=edit&redlink=1) on partial evaporation.

Treatment of chromium (III) sulfate with bases produces [chromium \(III\) hydroxide.](http://www.sciencemadness.org/smwiki/index.php?title=Chromium(III)_hydroxide&action=edit&redlink=1) If the base used is a carbonate or bicarbonate, this reaction is accompanied with the release of carbon dioxide, in a manner similar to iron, which does not form carbonates.

2.3 Physical Properties

Pure samples can be dried to form a lighter-colored powder, Chromium (III) sulfate most often appears as a blue-grey or violet-grey amorphous solid. Recently prepared samples or those that have been subjected to heat may contain a deep green, Chromium (III) sulfate is readily soluble in water and some lower alcohols.

2.4 Preparation

 Chromium (III) sulfates is a byproduct, along with sodium sulfate. If the temperature of the reaction is kept low enough, the presence of chromium (III) sulfate will be indicated by a blue or blue-violet color to the end product, rather than a green color produced when the temperature is too high. To extract the chromium (III) from solution, the reaction products must be treated with bases to precipitate chromium (III) hydroxide.

Chromium (III) sulfate can be more directly produced by the action of cold, dilute sulfuric acid on chromium (III) hydroxide or [chromium \(III\) oxide.](http://www.sciencemadness.org/smwiki/index.php/Chromium(III)_oxide) It should be noted, however, that many grades of chromium (III) oxide are not reactive enough, often as a result of being calcined, to respond to treatment with acid.

2.5 **Safety**

Contact with solutions of chromium (III) sulfate or inhalation of particles may cause irritation, but overall this compound is relatively non-toxic.

2.6 Storage

`Chromium(III) sulfate is relatively stable and has low reactivity, but should be kept away from temperatures greater than 40 °C when in the presence of moisture to avoid complex formation. A clean plastic bottle is a good storage bottle.

2.7 Disposal

Chromium (III) can be harmful to marine life, so water-soluble compounds of trivalent chromium should be precipitated as chromium (III) hydroxide before being disposed of in the trash. also recycle the chromium ions .[\(B2.2019\)](#page-63-2)

2.8 Chromium ore processing

There are two basic processes in metal processing:

1- Separation of valuable metals from mixed waste metals.

2 - The process of separating minerals of economic value of waste metals., [\(Wills, 1983\)](#page-64-7)

Cracking operations:-

The goal of the crushing process is to reach an appropriate size and the crushing is done in two stages:-

1- First cracking, to reduce the size of the raw particles to a suitable size for transportation and feeding secondary crushers:

2- Secondary cracking, It is used to reduce the resulting sizes to sizes suitable for grinding operations. The cone crusher is one of the most important secondary crushers and is

considered as a modified roll crusher. It receives ore size of 30 cm and produces sizes ranging from (2.5-3) cm .It has capacities up to 1100 tons per hour and is characterized by high speed [\(Al-Sabahi, 2009\)](#page-63-3)

Grinding Processes :The grinding is the last stage of crushing (size reduction) 'and is conducted on the crude resulting from crushing operations' and is obtained in the crude sizes ranging between (10-300) micron' The purpose of the grinding operations is to reach the crude to a suitable size for the concentration operations .

Gravity concentration: Here we will focus on the method of gravity concentration as it is the main method used in this research to raise the grade of low grade chrome ore. Gravity separation methods are used to process a wide range of materials ranging from heavy metal sulphides such as galena with a specific density of 7.5 to coal with a specific density Gravity separation methods are used to separate different density metals according to the relative difference in movement under gravity. There should be a significant difference between the density of the metal and the waste in order for the separation process to be good. Therefore, they are less responsive to gravity concentration, especially in the case of mass production.

Gravity Separators:

There are two types of Gravity Separators:-

1-Snail

The snail consists of connections in the form of circular segments.

2- Focus the vibrating table

When a thin membrane of water glides on a sloping surface, the surface of the water near the surface has a slow motion due to friction that occurs on the sloping surface and the velocity increases towards the surface of the water. In the slow part of the water membrane, high-density grains move more slowly than light grains, thus causing lateral displacement of materials.

The vibrating counter and other gravitational concentrations also require large amounts of water. Dry separation may sometimes be used, especially when water is difficult to obtain. It is used in the process of lateral separation of chrome ore to give the final position.**)** Al-Sabahi, 2009)

Concentration by flotation:

One of the most important and widespread methods of ore processing technology is the floating concentration method. This method is based on changing the surface conditions of the granules by auxiliary factors to make precious metals tend to air and waste metals tend to water and vice versa.

Product Chromites ore:

The crude is processed after crushing and grinding, then washed with stirring. after that float chromites ore by sodium fluorite and sulfuric acid as regulators , and emulsifier complex of pine oil, fuel oil and petroleum sulfonate under conditions of high solids and is formatted at pH 5 and thus obtain chromium oxide center by approximately (36-43%) by the recovery of 91 % ,[\(Alnsr, 2009\)](#page-63-4) .

Figure (2 .2) Chrome ore factory in the Damazin, Blue Nile State

Figure (2.3) Layout of the Ball Mill

2.9 Design devices that are used in the production work to produce BCS

2.9 Rotary kilns

Principle of operation:

The kiln is a cylindrical vessel, inclined slightly to the horizontal, which is rotated slowly about its longitudinal axis. The process feedstock is fed into the upper end of the cylinder. As the kiln rotates, material gradually moves down toward the lower end, and may undergo a certain amount of stirring and mixing. Hot gases pass along the kiln, sometimes in the same direction as the process material (co-current), but usually in the opposite direction (counter-current). The hot gases may be generated in an external furnace, or may be generated by a flame inside the kiln. Such a flame is projected from a burner-pipe (or "firing pipe") which acts like a large [Bunsen burner.](https://en.wikipedia.org/wiki/Bunsen_burner) The [fuel](https://en.wikipedia.org/wiki/Fuel) for this may be gas, oil, pulverized petroleum coke or pulverized coal.

Construction:

The basic components of a rotary kiln are the shell, the [refractory](https://en.wikipedia.org/wiki/Refractory) lining, support tyres (riding rings) and rollers, drive gear and internal heat exchangers.[\(B3.2019\)](#page-63-5)

 Figure (2.4) Rotary kiln for chrome

2.10 Design of CSTR

Introduction

A continuous stirred tank reactor (CSTR) is reactor equipped with an impeller or other mixing device to provide efficient mixing. In chemical engineering the name CSTR is often used to refer to an idealized agitated tank reactor used to model operation variables required to attain a specified output . Chemical reactors often have significant heat effects, so it is important to be able to add or remove heat from them. In a CSTR (continuous stirred tank reactor) the heat is added or removed by virtue of the temperature difference between a jacket fluid and the reactor fluid.[\(B4.2019\)](#page-63-6)

General Information:Continuous Stirred-Tank Reactors (CSTRs) are open systems, where material is free to enter or exit the system that operates on a steady-state basis, where the conditions in the reactor don't change with time. Reactants are continuously introduced into the reactor, while products are continuously removed.(CSTRs) are very well mixed, so the contents have relatively uniform properties such as temperature, density, etc. throughout. Also, conditions in the reactor's exit stream are the same as those inside the tank.

Equipment Design : CSTRs consist of a tank, usually of constant volume, and a stirring system to mix reactants together. Feed and exit pipes are present to introduce reactants and remove products.[\(B5.2019\)](#page-63-7)

Usage Examples: Continuous stirred-tank reactors are most commonly used in industrial processing, primarily in homogeneous liquid-phase flow reactions, where constant agitation is required. They may be used by themselves, in series, or in a battery. CSTRs are also used in the [pharmaceutical industry](http://encyclopedia.che.engin.umich.edu/Pages/Pharmaceuticals/menu.html) as a loop reactor. A dimple jacketed pressure vessel is shown in the picture on the left, and a half pipe coil jacket reactor is on the right .[\(B5.2019\)](#page-63-7)

Table (2.1): Productive recipe

The chemical analysis of chromites show that chromic oxide content in chromites is 43.66%

Chromic oxide is $= 43.66\%$

Particle size of chromium ore = 74 microns

Basic (chromites ore) =1000kg/h

Amount of chromic oxide in feed sample $= 0.4366*1000 = 436.6$

Amount of sodium carbonate in feed =700 kg/h

Molecular weight of $cr₂o₃ = 152$ kg/k mol

Moles of cr2o3 in feed =436.6/152 = 2.87 k mol/h

Roasting process for production of sodium chromate

1000kg/h of chromites ore powder was crushed to 74 micron, mixed with 700kg sodium carbonate in batch reactor kiln at 1200C in an oxidizing atmosphere, it is inside the kiln for about four hours until sodium chromate is formed. The product from the kiln was taken out and cooled.The cooled product then is leached in a large bottomed box by using water to dissolve the sodium chromate.

 $2Cr_2O_3 + 3O_2 + 4Na_2CO_3 \rightarrow 4Na_2CrO_4 + 4CO_2$ …………………………(2-1)

From The Reaction: 2moles of Cr_2O_3 produced 4 moles of chromate

Moles of chromate produced = $(48*2.87)/2=5.74$ kmol/h

Molecular weight of $Na₂Cr₉ = 162kg/k$ mol

1. Amount of chromate produced $=$ (moles of chromate produced) * (molecular weight of $Na₂Cro₄)$

 $= (5.74k \text{ mol/h})*(162kg/k \text{ mol}) = 929.88kg/h$

After the roast is cooled and transferred to leaching tank where water is added and a nearly saturated solution of sodium chromate is obtained with efficiency of 80%

Reaction of chromate to dichromate in CSTR:-

Produced chromate entered CSTR with sulfuric acid (98%) to produce dichromate by the reaction:

 $2Na_2CrO_4 + H_2SO_4 + H_2O \rightarrow Na_2Cr_2O_7 + Na_2SO_4 + 2H_2O$ ……………(2-2)

 The dichromate then entered the evaporated to increase the concentration of the dichromate from (80% to 85%)

2moles of chromate is reacted with 1mole of sulfuric acid.

Moles of sulfuric acid = $(5.74 \text{kmol/h})/2 = 2.875 \text{ k mol/h}$

Molecular weight of acid $= 98$ kg/k mol

Finally:-

Amount of sulfuric acid = $2.875*98 = 281.75$ kg/h

2moles of chromate is produced 1mole of dichromate

Moles of dichromate produced = $(5.74)/2 = 2.875$ k mol/h

Molecular weight of Na2cr2o7 = 262 kg /k mol

In the end a mound of dichromate can be produced

=2.875*262=753.25 kg/h

Reaction of dichromate to BCS in CSTR:-

Calculations Design of CSTR

General mole balance:-

In - Out + generation $=$ accumulation

$$
2Cr_2O_3 + 3O_2 + 4Na_2CO_3 \rightarrow 4Na_2CrO_4 + 4CO_2
$$
 (2-3)
\n
$$
2Na_2CrO_4 + H_2SO_4 + H_2O \rightarrow Na_2Cr_2O_7 + Na_2SO_4 + 2H_2O
$$
 (2-4)
\n
$$
4Na_2Cr_2O_7 + 12H_2SO_4 + C_6H_{12}O_6 + 26H_2O \rightarrow 8Cr(OH)SO_4 + 4Na_2SO_4 + 40H2O + 6CO_2
$$
 (2-5)

Table (2.2): Reaction feeds components properties

Intensity = M/V_0

 $V_0 = M/I$

Volumetric flow rate Na₂cro₄

V Na₂cro_{4 =} (929.88*1000) / (2.52*10⁶) = 0.369 (m³/h)

……………………………………………………………………..

Volumetric flow rate H2so4

 $V_{H2so4} = (281.75*1000) / (1.84*106) = 0.153 \text{ m}3/\text{h}$

 V_0 = Total volumetric flow rate

 $V_0 = 0.522$ m3/h

 $F_{A0} = F_{A0} (Na_2cro_4) + F_{A0} (H_2so_4)$

 $(929.88/162)$ + $(281.75/98)$ = 5.74 + 2.875 = 8.615 K mol/h

$$
C_{A0} = F_{A0} / V_0
$$

(2-6) = 8.615/0.522=16.5

$$
J = v/v_0 \quad (1.58/0.522) = 3.03
$$

\n
$$
J = (C_{A0}X) / (-RA) \quad , -RA = KC_{A0} (1-X), J = X/(K(1-X))
$$

\nAssume:
\n
$$
X=90\% \quad , J = 3h
$$

\n
$$
3=0.9/(K(1-X)) \quad , k = 3h^{-1}
$$

\n
$$
-RA = 3*16.4(1-0.9) = 4.92
$$

\n
$$
8.615/0.522=16.5
$$

\n
$$
V_{CSTR} = (F_{A0}*X) / -RA \quad , V_{CSTR} = (8.615*0.9)/4.92 = (7.75/4.92) = 1.58 \text{ m3}
$$

Determination of diameter and height :

We can take the ratio $h/D = 3$ and substitute this ratio in this equation

 $V=\pi/4D2h$ $(2-7)$

 $V = \pi/4D2*3*D = \pi/4*3*D3 = 1.58 m3$ $D_3 = (1.58)/(\pi/4 \cdot 3) = 1.58/2.4 = 0.658, D = 0.87$ $h = V/(\pi/4*D2),$ h=1.58÷(0.79*(0.87)2) h= $1.58 \div 0.597 = 2.65$ m 80% = real quantity of the solution in the reactor $D_{ac} = D/0.8 = (0.87/0.8) = 1.09$ m

H _{ac} = $h/0.8 = (2.65/0.8) = 3.313$ m

Vessel thickness :

 According to the British standard, BS-5500 the minimum thickness of a cylindrical vessel under internal pressure depends on the internal diameter of the vessel, the internal pressure and the designed stress when ignoring the welded

Table (2.3): Summary of design of CSTR

Figure (2.5)Cross-sectional diagram, for CSTR Reactor.[\(B6.2019\)](#page-63-8)

2.11 Evaporation

 Evaporation, one of the main methods used for the concentration of aqueous solution, refers to the removal of water from a solution by boiling the liquor in a suitable vessel, the evaporator ,and withdrawing the vapor .If the solution contains dissolved solids , the resulting strong liquor may become saturated so that crystals are deposited liquors. Evaporation is carried out by adding heat to solution to vaporize the solvent .The heat is supplied principally to provide the latent heat of vaporization ,and ,by adopting methods for recovery of heat from the vapor ,it has been possible to achieve great economy in heat utilization .Whilst the normal heating medium is generally low pressure exhaust steam from turbines ,special heat transfer fluids or flue gases may be used .The design of an evaporation unit require the practical application of data on heat transfer to boiling liquids ,together with a realization of what happens to the liquid during concentration

Evaporator Units: There are two main types of evaporators units; Single Effect and Multiple Effect Evaporators. Single-effect evaporators are used when the throughput is low, when a cheap supply of steam is available, when expensive materials of construction must be used as is the case with corrosive feed stocks and when the vapor is so contaminated so that it cannot be reused. Single effect units may be operated in batch, semi-batch or continuous batch modes or continuously.

A multiple-effect evaporator is an evaporator system in which the vapor from one effect is used as the heating medium for a subsequent effect boiling at a lower pressure. Effects can be staged when concentrations of the liquids in the effects permit; staging is two or more sections operating at different concentrations in a single effect.

Evaporator Design: Three principal elements are of concern in evaporator design: heat transfer, vapor-liquid separation, and efficient energy consumption. The units in which heat transfer takes place are called heating units or Calendars'. The vapor-liquid separators are called bodies, vapor heads, or flash chambers. The term body is also employed to label the basic building module of an evaporator, comprising one heating element and one flash chamber.

The system selected is a quadruple effect evaporator system used for concentration of saline water. Falling film evaporator is used for this system with forward flow sequence.

Operating parameters for this system are mentioned below in the Table:

Table (2.4): Operating Parameter for quadruple system:

Design Model

In this section we present our design calculations in three subsections; design steps, design model and calculations.

A- Design Steps

- First calculate of overall mass balance
- Calculate of temperature in each effect by $Q = u_1 A_1 \Delta T_1 = u_2 A_2 \Delta T_2 = u_3 A_3 \Delta T_3$

Assuming $A_1=A_2=A_3$

 $\sum \Delta T = \Delta T_{1+} \Delta T_{2+} \Delta T_3$ \sum ΔT=ΔT_{1+ $\frac{u_1}{u_2}$ ΔT1_{+ $\frac{u_1}{u_3}$ ΔT1}}

- Calculate of energy balance by solve these equations to obtained of steam in each effect by; $D_1+D_2+D_3=0.95$

 $D_0^* \lambda_0 = m_f^* C_f (T_1 - T_f) + D_1^* \lambda_1$

 $D_2^* \lambda_2 = (m_f - D_1)^* c p_1 (T_1 - T_2) + D_1^* \lambda_1$

 $D_3^* \lambda_3 = (m_f - D_1 - D_2)^* c p_2 (T_2 - T_3) + D_2^* \lambda_3$

- Concentration per effect is calculated by this equation from figure 1;component balance:

M^f =D1+mp1……………………. (2-9) $M_f * X_F = D_1 * x_{D1} + m_{p1} * x_{p1}$ ………… (2-10)

xD1=0…………………………… (2-11) MF*XF=mp1*xp1……………….… (2-12)

From e.g. (1):

 m_{p1} = m_F - D_1

 $MF^*X_F=(m_f-D_1)^*x_{p1}$ ……………….…. (2-13)

Similarly for effect 2 and 3:

xp2 ………………... (2-14) xp3 ……. (2-15)

- Calculate of mass balance around each effect to obtained of water condensation for each effect
- Calculate amount of steam economy by: $SE = \frac{mE}{ms}$
- Then after area of each effect is calculate by: $A = \frac{b \theta \cdot \lambda_0}{(U + \Delta T)}$
- No of tubes are found by: $Nt = \frac{A}{(n \cdot D \cdot L)}$

B- Model Diagram

Triple Effect Evaporators is an evaporator system in which the vapor from one effect is used as the heating medium for a subsequent effect boiling at a lower pressure.

Figure (2.6): triple Effect Evaporators

Evaporators Types: There are many types of evaporators which are named below:

- Horizontal Tube Evaporators
- Horizontal Spray Film Evaporators
- Long Tube Vertical Evaporators
- Short Tube Vertical Evaporators
- Basket Type Evaporators
- Forced Circulation Evaporators
- Agitated thin Film Evaporators or wiped film evaporator
- Plate Evaporators

Method of feeding: Depending upon the directions of flow of the heating medium and of feed of the liquor.

Multiple effect evaporators can be classified in to four categories :

- 1. for word feed
- 2. Back word feed
- 3. Mixed feed
- 4. Parallel feed

Evaporators are often classified as follows:

- Heating medium separated from evaporating liquid by tubular heating surfaces.
- Heating medium confined by coils, jackets, double walls, flat plates, etc.
- Heating medium brought into direct contact with evaporating liquid.
- Heating with solar radiation.

Evaporators with tubular heating surfaces dominate the field. Circulation of the liquid past the surface may be induced by boiling (natural circulation) or by mechanical methods "forced circulation". In forced circulation, boiling may or may not occur on the heating surface.

2.12 Design of spray dryer:

Introduction

Spray drying is a one-step continuous unit operation that employs liquid atomization to produce droplets that are dried to individual particles when moved in a hot gaseous drying medium. A spray dryer consists of a feed pump, atomizer, air heater, air dispenser, drying chamber and systems for exhaust air cleaning and powder recovery/separator.

The three stages that occur in a spray dryer before drying is accomplished include:

- 1. Atomization
- 2. Spray-air mixing and moisture evaporation.
- 3. Dry product separation from the exit air.

The nature of the final product obtained after drying in a spray dryer depends on:-

- A. The design and operation of the spray dryer.
- B. The physicochemical properties of the feed.

Spray dryer design considerations: The most important variables in the design of a spray dryer are the evaporation rate and the required particle size distribution of the product. The evaporation rate and the dryer ΔT (inlet air temperature minus the outlet air temperature) dictates the amount of drying air needed, which dictates the sizing and cost of almost all of the system components. The particle size requirement affects the choice of atomization and can also affect the size of the spray dryer.

For a given evaporation rate, the higher the design ΔT , the less process air is required, and the smaller and less costly the spray drying system will be. Many products, though, cannot use high inlet temperatures due to the potential of thermal degradation of the product . [\(B7.2019\)](#page-63-9)
Dryer Calculations:-

Figure (2.7) spray dryer Diagram

Figure (2.8) FLOW Diagram for production of Basic chromium Sulphate

Notice:

When you want to use basic chromium sulphate in apowder form, it was followed below :

- 1. Evaporator
- 2. Spray dryer
- 3.Packaging

2.13 The Chemistry of Mineral Tannage

Delimed pelt- outcome of pre-tonnage is still raw material. When moist it is soft and pliable. The aim is to make it durable, soft, porous opaque, together with stability over a wide range of physical and chemical conditions (pH, T, humidity).

The tanning agent must be able of cross linking the molecules of collagen (has to be multifunctional). Degree of cross linking needs careful consideration: if too much crosslink, than the product is harsh and brittle (mobility of fibrils are restricted).

Apart from introducing a limited number of cross links, a tanning agent should not at the same time lead to undue fibril modification (reduction in fibril length or solution of protein material). In addition to number of cross links introduced by tanning, their general character is of importance. Could be: H-bonds, ionic bonds, covalent bonds.[\(B8.2019\)](#page-63-0)

2.14 Chemical Control in Chrome Tanning

2.14.1 Basicity:

All types of chrome compounds cannot tan protein it must have hydroxyl group in the complex directly attached to the chromium atom, such compound are called basic compound.Tanning takes place with Basicity between 33% and 55.above 55% Basicity there is danger of chrome precipitation and subsequent staining of the hide. Chromium sulphate in solution has strong attraction for hydroxyl ions oh which associate with molecule by replacing one or more of the sulphate ions $so₄$. As more (OH) is made available, (i.e., as the pH rises),it enters the chromium sulphate molecule and the molecules reactivity toward collagen increases. However when the pH rises beyond about pH 4.3 all the sulphates radicals are removed and chromium hydroxide is formed Chromium hydroxide is insoluble and has no tanning powder at all. It is useful to note that below pH 4 the amount of acid used in pickling will need and equivalent amount of alkali to add for basification. Chromium sulphate Cr_2 (SO₄)₃ itself has only a slight tanning effect. Considerably better results are obtained with (Basic chromium Sulphate) in which some of the sulphate radicals are replaced by hydroxyl radicals. This tanning effect, often called the astringency, increases with the number of (OH) groups until a Basicity of approximately 66% is reached, when the chromium compounds become insoluble and precipitate. The Basicity is expressed in % Schorlemmer and indicates the percentage e of valences in the chromium (III) compounds which has been replaced by (OH) groups (6)

Table (2.6): Basicity of Chromium (III) Salphate

2.14.2 Olation

 The bonds between hydroxyl and two chromium atoms , Is the following reaction in which a complex consists of hydroxyl and 2 chromium atoms and these hydroxyl does not react with acids in the titration process, With the increase of the hydroxyl group increases the basic and trend in non-thawing . Basic chromium salphate undergo Olation if left to stand in an aqueous solution. In fact it is this process which turns a chromium compound into a chrome tanning material. In Olation the OH groups in the basic chromium sulphate react with another molecule by means of H- bonding to give higher molecular weight chromium compounds, as shown below:[\(B9.2019\)](#page-63-1)

2.14.2.1 Olated polynuclear complexes:

Here the metal ions are linked through OH bridges

^{……………………. (2-22)}

Rate of reaction will depend on the nature of the ligands already present in the chrome complex. If large amounts of very stable masking ligands such as oxalate ions are present, no tannage will occur, since these cannot be displaced by carboxyl groups , Little is known, of the rate of reaction between particular chromium complex ions and competing ligands, whether in solution or in actual tannage of pelt. Following reaction sequence suggested:

Conditions are likely to be most complex, overall reaction rate being influenced by factors such as:

- a) Compactness of pelt structure (affect diffusion rates)
- b) Sizes of Cr complexes
- c) Rate of over-all-coordination
- d) PH
- e) Temperature
- f) Relative concentration of reactants
- g) Nature of ligands in complexes
- h) nature and addition sequence of competing ligand .[\(B8.2019\)](#page-63-0)

CHAPTER TWO

Section Two

The impact of Basic Chromium Sulphate on the Environment

2.15 Chromium exposure study in chemical based industry

Results and Discussion the results of chromium exposure :-

Environmental monitoring: The levels of Cr were found higher at basic chromium salphate (BCSu) (45.87 \pm 20.4 µg/m3), dichromate plants (33.08 \pm 13.15 µg/m3), followed by furnace $(22.49 \pm 11.18 \text{ µg/m3})$, filtration $(22.48 \pm 11.25 \text{ µg/m3})$, spray dyer $(22.43 \pm 10.7 \text{µg/m3})$ and office/lab $(4.21 \pm 0.61 \mu g/m3)$. The Cr levels were observed lowest in office and laboratory areas. The overall results show that Cr levels in the plant were observed lower than the threshold limit value (i.e. $500 \mu g/m3$). Cr (VI) compounds are emitted into the air, water, and soil by a number of different industries. In the air, chromium compounds are present mainly as fine dust particles that eventually settle over the land and water . In a recent review, (Feldman[,etal, 2004\)](#page-64-0) a chromium exposure up to about 5000 µg/m3 in the chromium plating industry was mentioned, but most exposure levels reported were in the range of 100-200 µg/m3, [\(NIOSH, 1973\)](#page-64-1). In modern plants, values are often less than 10 µg/m3,previously reported normal levels of chromium in blood of 2-3 µg/100ml,[\(Feldman,](#page-64-2) [1967\)](#page-64-2) have probably been too high. At present the concentration of chromium in plasma has been reported to be 0.014 μ g/100 ml. In Encyclopedia of occupational Health and Safety, 4th edition (1998) published by the international labour office has mentioned the values in serum and urine does not exceed 0.05 µg/100ml and 2.00 µg/g creatinine, respectively.In conclusion, there is no clear evidence to relate exposure to environmental levels of chromium with adverse health effects in the general population or subgroups exposed to chromium around industrialized sites. Based on results of ambient air monitoring and biological monitoring, following recommendations are made to minimize chromium exposure to workers at work places and to protect the health of individuals.the help of better housekeeping and better industrial hygiene practices. Workers employed for sweeping, cleaning, distillation, ore mixing of chromium and furnace workers should be provided personal protective device like certified masks, aprons, gloves etc. during the work shift. All the workers/staff should be made aware of health hazards due to chromium and its salts. Practice of personal hygiene may help to reduce the exposure risk due to chromium. It is necessary to organize workers education and awareness campaign. There is

a need for periodical air monitoring (at least every three-month) and medical surveillance including biological monitoring (every year) of exposed workers of industry.

2.16 Chromium in the environment.

These are not usually allowed to exceed the following limits:-

* Total dust 20 mg/ m³ Chromium . Trivalent Chromium 5mg/m³, Hexavalent Chromium 1 mg/m^3 .

* for liquid effluent the concern is over the amount of chromium in the suspension in the released fluids and this is monitored at the points of discharge and seepage of the effluent. Usual limits for this purpose are:-

Total chromium 0.5mg / Liter . Hexavalent Chromium 0.1 mg/ Liter .

* For solid wastes the fact that they contain chromium is sufficient to render them as "dangerous" in most countries and consequently these should be treated with extreme care under strict regulations. As a general measure, however industry is encouraged to keep production of solid wastes to a minimum and, if necessary limit waste disposal to within the plant itself or minimize its transportation. Another measure is of course, is to recycle any waste, whenever is possible .[\(B10.2019\)](#page-63-2)

2.17 Human Health Effects:-

Human Exposure and Toxicity:

* Chronic exposure can produce contact dermatitis, induced pemphigus.

* Bronchial reactivity can be specifically induced by fumes of chromium sulfate.

* Fatal case was described, when a woman ingested 400 ml of leather tanning solution containing 48 g of basic chromium sulfate.

* Occupational exposure to trivalent chromium can lead to DNA damage.

Animal Studies: The toxicity of basic chromium sulfate was investigated in rats in a 13 week nose-only inhalation study that included a 13-week recovery period. Basic chromium sulfate produced severe and widespread effects in the nasal cavity, larynx, lungs, and mediastinal lymph node. Effects were characterized by accumulation of foreign material, infiltration of alveolar macrophages, septal cell hyperplasia, and granulomatous and chronic inflammation. As little as 10g or Cr(III) (as chromic sulfate) elicited allergic responses in sensitized guinea pigs.[\(B11.2019\)](#page-64-3)

2.18 Environmental Standards & Regulations:-

RCRA Requirements:

A solid waste containing chromium may or may not become characterized as a hazardous waste when subjected to the Toxicity Characteristic Leaching Procedure listed in 40 CFR 261.24, and if so characterized, must be managed as a hazardous waste. [\(USEPA, 2015b\)](#page-64-4)

Clean Water Act Requirements:

Chromic sulfate is designated as a hazardous substance under section $31(1)(b)$ (2)(A) of the Federal Water Pollution Control Act and further regulated by the Clean Water Act Amendments of 1977 and 1978. These regulations apply to discharges of this substance. This designation includes any isomers and hydrates, as well as any solutions and mixtures containing this substance.[\(USEPA, 2015b\)](#page-64-4)

2.19 Cleanup Methods:-

2.19.1 Accidental Release Measures:

 Personal precautions: Protective equipment and emergency procedures: Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust.

Environmental precautions:

 Do not let product enter drains. Materials for containment and cleaning up: Pick up and arrange disposal without creating dust. Keep in suitable, closed containers for disposal.[\(Sigma-Aldrich, 2015a\)](#page-64-5)

* If chromium metal or insoluble chromium salts are spilled, the following steps should be taken:

1. Remove all ignition sources where metallic chromium has been spilled.

2. Ventilate area of spill.

3. Collect spilled material in the most convenient and safe manner and deposit in sealed containers for reclamation or for disposal in a secured sanitary landfill.

Liquid containing chromium metal or insoluble chromium salts should be absorbed with vermiculite, dry sand, earth, or a similar material.[\(Mackison, 1981a\)](#page-64-6)

2.20 Disposal Methods:

Contact a licensed professional waste disposal service. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator

.[\(Sigma-Aldrich, 2015b\)](#page-64-7)

Generators of waste (equal to or greater than 100 kg/mol) containing this contaminant, USEPA hazardous waste number D007, must conform with USEPA regulations in storage, transportation, treatment and disposal of waste. [\(USEPA, 2015a\)](#page-64-8)

Waste water from contaminant suppression, cleaning of protective clothing/equipment, or contaminated sites should be contained and evaluated for subject chemical or decomposition product concentrations.

Concentrations shall be lower than applicable environmental discharge or disposal criteria.

If it is not practicable to manage the chemical in this fashion, it must be evaluated in accordance with EPA 40 CFR Part 261, specifically Subpart B, in order to determine the appropriate local, state and federal requirements for disposal. [\(USEPA, 1982\)](#page-64-9)

2.21 Manufacturing/Use Information:

Insolubilization of gelatin; in catalyst preparation; as mordant in textile industry; in tanning of leather; in chrome plating; in manufacture of Cr /chromium/, CrO3 /chromic trioxide/, and Cr alloys; to improve dispersibility of vinyl polymers in water; in manufacture of green varnishes, paints, inks, glazes for porcelain.

Table (2.7) Chromium compounds.

[\(al, 2000\)](#page-63-3).[\(Mackison, 1981b\)](#page-64-10)

CHAPTER THREE

MATERIALS AND METHODS

3.1. Study location

This study was conducted in the of Chemistry Laboratory in Leather Industries Incubator And also the chemistry lab, Department of chemical engineering, College of Faculty of Engineering and Technology of Industries, Sudan University of Science and Technology The experiment was also conducted at the Center Research and Consulting for Industrial , the General Authority for Geological Research and the central oil laboratories in cooperation with the National Center for Leather Technology, Advanced Mining Works Company in Blue Nile State ,during (August - December, 2019).

3.2. Materials, tools and equipments used in the study or Reagents and materials

3.2.1 Materials:-

3.2.3 Tools and equipments:

3.3 Methodology

3.3.1 Collection of samples

Samples of chromium were collected from an advanced mining company in Blue Nile State, as obtained Samples of sodium carbonate, calcium carbonate, glucose, sodium hydroxide and other chemicals and tools used in the experiment from New Life Center in Khartoum.

3.3.2 Experiments

3.3.3 Preparation of the tested samples

1. The integrity and quality of the sample components were maintained at all stages of manufacturing.

2. Beginning from collection, preparation and analysis, labels reflect the content and details of the sample.

3. I grind the coarse samples for easy mixing and then react with the mixture materials.

3.3.4 Oven heating

Mixed the chromites brought from the advanced mining company , In the Blue Nile state the percentage of chromium oxide was 43.66% with sodium carbonate with a purity of 98%. Calcium carbonate was added, roasted in the rotary kiln, the temperature was set at 1200 ° C and oxygen was allowed to pass. On the first day the heating was done for two hours and the sample was left for the over night.

Note: The color of the granules has been changed to yellow. On the second day the oven was turned on for four hours continuously, stirring every half hour.

Note:-

A change in the color of the mixture to yellow was observed. Monosodium chromate was then obtained.

3.3.5 Roasting of the chromites ore with Na2CO3

Iron in chromites roast reacts with sodium carbonate to produce NaFeO2+NaAlSiO4 and other complex sodium-aluminum silicate according to the following reaction .

$$
2FeCr_2O_4 + 1/2O_2 \rightarrow 2Cr_2O_3 + Fe_2O_3 \quad (3-1)
$$

\n
$$
2Cr_2O_3 + 2Na_2CO_3 \rightarrow 2Na_2Cr_2O_4 + 2CO_2 \quad (3-2)
$$

\n
$$
2Cr_2O_3 + 3O_2 + 4Na_2CO_3 \rightarrow 4Na_2CrO_4 + 4CO_2 \quad (3-3)
$$

\nHence, the overall reaction can be expressed as follows:
\n
$$
4FeCr_2O_4 + 8Na_2CO_3 + 7O_2 \rightarrow 2Fe_2O_3 + 8Na_2CrO_4 + 8CO_2 \quad (3-4)
$$

3.3.6 Extraction stage

1. At the beginning, the resulting mixture from the roasting process was grinded

2. The mixture was dissolved in tanks of water and the pH (8) was adjusted, the filtration of the mixture was carried out to separate the sodium chromites liquid from the other precipitates.

3 - Process of purification of sodium chromites; the solid residue was separated quantitatively and digested.

4- Sodium chromate was not recovered but instead converted it directly to sodium dichromate after adding 1 ml sulfuric acid, 10 g sodium chromate, 16 ml water.

5. The solution is treated with enough sulfuric acid to convert chromate into dichromate. As a result, sodium sulphate is formed and most sodium sulphate crystallizes in the aqueous state from the hot boiling solution during roasting. The rest falls into the vaporizers while the dichromate solution is concentrated.

3.3.7 Procedure for preparation of Chromium basic sulphate:-

Dissolve the mixture in water tanks, raise pH and produce monosodium sodium chromate which is deposited and dried.

Addition of sulfuric acid to a mixture of monosodium chromate and production of sodium dichromate.[\(works, 1995\)](#page-64-11)

3.3.8 Stage of Preparation of basic chrome sulphate

The liquors containing $Na₂CrO₄$ obtained in the previous experiments were acidified with sulfuric acid to form $Na₂Cr₂O₇$ according to the following equation (3-3)

 $2Na_2CrO_4 + H_2SO_4 + H_2O \rightleftharpoons Na_2Cr_2O_7 + Na_2SO_4 + 2H_2O$ (3-5)

Basic chrome sulfate can then be manufactured by simple reduction of the resultant Na2Cr2O7 in acid media to basic chrome sulfate .

3.4 Reduction of sodium Dichromate to basic chromium sulphate:

250 gram of sodium dichromate was taken from leached chromium 6 in a beaker :230 g of concentrated sulfuric acid (Sp.Gr.1.84) were added carefully and stirred well .the beaker was placed in a reactor under coaling with continuous stirred while 62.5 g of sugar were added slowly in 10 lots over three hours . During the reaction the color of the liquor changed gradually from orange to green and finally to bluish green indicating the completion of the reduction. The reaction proceeded as follows:

 $4NaCr_2O_7+12H_2SO_4+C_6H_{12}O_6+26H_2O \rightarrow 8Cr(OH)SO_4+4Na_2SO_4+40H2O+6CO_2$ (3-6) [\(SHARAF, 2013\)](#page-64-12)

3.5 Determination of Basicity:

Basicity computation is calculated from the following law

Basicity = ((A-B)/A) ×100 ……… ……………………………… (3-7)

A= ml of 0.1 normal sodium thiosulphate.

B= ml of 0.1 normal sodium hydroxide

3.6 Chemical analysis of leather produced:

Also, the following tests must be carried out on samples of leather that are tanned with chromium produced to ensure the quality of the base chromium sulfate produced.

(% chromic oxide, % total ash, % insoluble ash)

CHAPTER FOUR

RESULT AND DISCUSSION

4.1 Experiment:

Chrome samples from Blue Nile State of Jam area

Table (4.1) preheating stage:

Photo (4.1) Sample mixture of chromites ore, sodium carbonate, and calcium carbonate after heating

Noticeable:

Sodium chromate composition

Table (4.2) (A) Dissolving and filtering stage

 Photo (4.2) Monosodium chromate solution (Na2CrO4)

 Photo (4.3) Sodium dichromate solution (k2cr2o7)

 Photo (4.4) Basic chromium sulfate solution (Cr (OH) SO4**)**

Table (4.4) Leather process

4.2 Calculation of the Basicity:

1.5ml of 1% phenolnaphthalein solution was added to 150ml distilled water in a large porcelain bowel and adjusted to pink color with three drops of 0.1N Sodium hydroxide solution. 12.5 ml of solution was prepared as described above and titrated with continuous stirring at the boil to effect further hydrolysis of the chromium salt, with 0.1 N sodium hydroxide until the distinct pink coloration was obtained .

Basicity = ((A-B)/A) ×100 ……………………………………………… (4-1)

A= 3 mL of 0.1 sodium thiosulphate.

B=2 mL of 0.1 sodium hydroxide.

 $((3-2)/3)*100$

 $=(1/3)*100 = 33.33 %$

4.3 Determination of chromic oxide ratio:

- (1)12ml of chrome liquor was transferred into a conical flask and diluted with 50 ml distilled water.
- (2) 2N caustic soda solution was added drop by drop until the precipitated chromium hydroxide formed was just dissolved.
- (3) 12 ml of hydrogen peroxide solution (3%) was added.
- (4)Some boiling stones were then introduced into the vessel and the content were allowed to boil.
- (5)A small funnel was suspended in the neck of the conical flask to avoid any loss due to bubbling. The boiled solution (yellow chromate) was cooled and made up to the mark.
- (6)25 ml of the yellow chromate solution was pipette into an iodine flask , use points from sulfuric acid
- (7)Followed immediately by 5 ml of 10 % potassium iodide, the closed flask was left in a dark for 10 minutes.
- (8) the liberated iodine was titrated with 0.1 N sodium thiosulphate using freshly prepared starch as indicator , 2ml of starch was added to the iodine flask to get blue color solution ,the disappearance of the blue color indicated the end point , titrate value was noted .

4.4 Calculations:

1 ml of 0.1 N sodium thiosulphate $= 2.533$ mg chrome oxide

% Cr_2O_3 in leather = (ml 0.1 sodium thiosulphate solution)*100/g leather taken …………..…… (4-2)

 $\%Cr_2O_3$ = (Volume ml of 0.1N sodium thiosulphate solution $\times 0.002533$) ×100% / (Weight of sample leather)…………………….. (4-3)

Volume ml of $0.1N$ sodium thiosulphate solution = 3 ml

Weight of sample leather $=2 g$

% $Cr2O3 = 3.8$ %

4.5 Determination of total ash content of the leather

A small piece of leather was cut and made into small pieces. A crucible was taken, dried and weighted was done after placing the leather pieces in the crucible .it was heated gently first and when the leather is completely in a muffle furnace at 800C .The aching was completed in a muffle furnace at 800C .until the carbon was consumed. Then it was cooled in desiccators and waited till constant and weight was obtained.

Calculation: Weight of original leather sample taken $= 6g$

Weight of ash $= 0.135$

% total ash content = (Weight of ash / Weight of original sample) \times 100

% total ash content = $(0.135/6) \times 100 = 0.0225 \times 100 = 2.25$ %

4.6 Determination of the insoluble ash of the leather

Dry the leather sample on the filter paper cone in an air oven to remove the excess moisture and incinerate in a previously weighted (w1) crucible And then heat at($800+25$ ^oC in a muffle furnace until completely ached cool the crucible with ash in desiccators and weighted (W2) %insoluble ash = $(W1-W2) \times 100$ $W1=5$ $W2 = 0.135$

% insoluble ash = (5- 0.135) \times 100 = 4.865 %

4.7 Chemical analysis of leather produced:

Shrinkage temperature, chromic oxide, total ash . And the result showed acceptable properties compared with standard results as shown in table

Table (4.5) chemical analysis of the leather produced:

Item	Values
% Basicity	33.33%
% chromic oxide	3.8%
%total ash	2.25%
% insoluble ash	4.865%

4.8 Discussions

This research was carried out in the initial stages in the laboratories of the Sudan University of Science and Technology, and conducted the first experiment on raw chromium , brought it from blue Nile state and used oil labs to analyze the samples and prepare the results , then controlled the temperature of the oven, as well as the proportions of the mixture, until you get raw Mono sodium chromate with high concentration and quality in standard production time of two hours , At a temperature of 1200° C, a correlation was observed between the concentration of chromites and obtain the best result

According to Table No (4.2) a certain weight of sodium chromate was taken and a certain volume of water at a temperature between (90 -95) I filtered and extracted the sodium chromate solution that was titrated with concentrated sulfuric acid.

According to the schedule (4.3), the sodium dichromate was obtained first, and with the increase in the amount of acid and the use of glucose as a reducing agent, obtained a solution of the base chromium sulfate of bluish green color in a very good time.

The rule was set according to the law shown on page (46), so its value was 33.33, which is the appropriate rule for tanning in chrome.

After that moved to the product testing stage and brought one kilogram of the treated skin first and it was in the lime stage, and performed a process of removing lime and embalming and set the pH at 3 which is the appropriate to enter the chrome for the skin, and after adding the chrome installed it using formic acid, It was completely confirmed that the chrome entered the skin, all of which are shown in Table No (4.5)

And then, chemical tests were carried out on the skin that tanned using the produced chromium base sulfate and determined the percentage of chromium oxide as well as the total percentage of ash and the insoluble materials in the ash according to Table No (4.5), The ratios were also estimated according to Table No. (1), the design of the CSTR reactor, and a dryer for large and productive quantities.

Chapter five

Conclusions and Recommendations

5.1 Conclusions

 The process of manufacturing basic chromium sulfate consists of four main stages, which are the preparation and preparation of raw materials, the production of monosodium chromate, the production of bilateral sodium chromate and the production of basic chromium sulfate.

Basic chromium sulfate containing trivalent chromium has been recognized as toxic compared with the hexavalent from which is more toxic and carcinogenic.

The Sudanese chromites is considered one of the good raw materials, the specifications of which correspond to the specifications of the international raw materials used in various industries, knowing that the geological reserves are estimated at about 1.5 million tons.

These quantities available to us in Sudan, if given an added value, will provide the country with hard currencies and sufficient tanneries for basic chromium sulfate.

5.2 Recommendations

1. This scheme should be transformed into devices of larger sizes and productivity in order to contribute to increasing the GDP.

2. When designing a chromium sulphate plant, a control system is required to increase production quality and prevent gas leakage.

3. Take advantage of all research aimed at this aspect and confines them according to their specialization all to make a successful plan for a real chromium industry in Sudan.

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THE APPENDICES

World chromite production 2014 29m tonnes

 Figure (A.1)

 Figure (A.3)

Figure (A.4) 1-Ball Mill- Highly Efficient Grinding And Milling Machine

Figure (A.5) Ball mill The cement raw material blending process and its control system.[\(AP1.2019\)](#page-63-4)

Figure (A.6) Typical mill power consumption for various degrees of fineness . Actual values vary according to mill system efficiency and clinker hardness.[\(AP2.2019\)](#page-63-5)

Figure (A.7) Schematic diagram of ball milling process [\(AP3.2019\)](#page-63-6)

Figure (A.8) Some safety tools

TABLE (2.3).LIST OF COMMERCIALLY PRODUCED SECONDARY CHROMIUM CHEMICALS AND THEIR GENERAL USES 1, 14, 18

(International, 1982 [International, 1982 \)](#page-64-13)

11.2 EVAPORATOR DESIGN

Three principal elements are of concern in evaporator design: heat transfer, vapor-liquid separation, and efficient energy consumption. The units in which heat transfer takes place arecalled heating units or Calandra's. The vapor-liquid separators are called bodies, vapor heads, or flash chambers. The termbody is also employed to label the basic building module of an evaporator, comprising one heating element and one flash chamber.

The system selected is a quadruple effect evaporator system used for concentration of saline water. Falling film evaporator is used for this system with forward flow sequence.

Operating parameters for this system are mentioned below in the Table 1:

Table1: Operating Parameter for quadruple system:

2.11.3 DESIGN MODEL

In this section we present our design calculations in three subsections; design steps, design model and calculations.

C- Design Steps

- First calculate of overall mass balance
- Calculate of temperature in each effect by $Q = u_1 A_1 \Delta T_1 = u_2 A_2 \Delta T_2 = u_3 A_3 \Delta T_3$ Assuming $A_1=A_2=A_3$

 $\sum \Delta T = \Delta T_{1+} \Delta T_{2+} \Delta T_3$ \sum ΔT=ΔT_{1+ $\frac{u_1}{u_2}$ ΔT1_{+ $\frac{u_1}{u_3}$ ΔT1}}

- Calculate of energy balance by solve these equations to obtained of steam in each effect by; $D_1 + D_2 + D_3 = 0.95$

 $D_0^* \lambda_0 = m_f^* C_f (T_1 - T_f) + D_1^* \lambda_1$ $D_2^* \lambda_2 = (m_f - D_1)^* cp_1(T_1 - T_2) + D_1^* \lambda_1$ $D_3^* \lambda_3 = (m_f - D_1 - D_2)^* c p_2 (T_2 - T_3) + D_2^* \lambda_3$

- Concentration per effect is calculated by this equation from figure 1;component balance:

Similarly for effect 2 and 3:

 $x_{p2} = \frac{m f \cdot x F}{(m f - D1 - D2)} \dots (11)$ $x_{p3} = \frac{m f \cdot x F}{(m f - D1 - D2 - D3)} \dots \dots \dots \dots (7) \dots (12)$

- Calculate of mass balance around each effect to obtained of water condensation for each effect
- Calculate amount of steam economy by: $SE = \frac{mE}{ms}$
- Then after area of each effect is calculate by: $A = \frac{D\sigma \cdot \lambda_0}{(U \cdot \Delta T)}$
• No of tubes are found by: $Nt = \frac{A}{(n \cdot D \cdot L)}$

D- Model Diagram

A triple Effect Evaporators is an evaporator system in which the vapor from one effect is used as the heating medium for a subsequent effect boiling at a lower pressure.

Figure 1: triple Effect Evaporators Table 2: Mass Balance

A- Calculations

In this section we calculate temperature in each effect as in the following Equion's;

$$
U1 = 2.5KW/m_2.K, U2=2KW/m_2.K; U3=1.6KW/m2.K
$$

\n
$$
\sum \Delta T = \Delta T_{1+} \Delta T_{2+} \Delta T_{3}=40^0C
$$

\n
$$
\sum \Delta T = \Delta T_{1+} \frac{u_1}{u_2} \Delta T 1 + \frac{u_1}{u_3} \Delta T 1
$$

\n
$$
\Delta T1 = \frac{40}{1+\frac{2.5}{2}+\frac{2.5}{1.6}} = 10.50^0C
$$

\n
$$
\Delta T2 = \frac{U1}{U2} \Delta T 1
$$

\n
$$
\Delta T2 = \frac{2.5}{2} * 10.50 = 13.125^0C
$$

 $T_1 = T0 - \Delta T1$ T1=110-10.50=99.5**⁰C** $T_2 = 86.4$ ⁰**C**

• From steam table:

Table 3: Steam Table Parameters

T °C	λ kcal/kg
$T0 = 110$	λ0=2230.038
$T1 = 99.5$	λ 1=2256.816
$T2 = 86.4$	λ 2=2292.444
$T3=70$	λ3=2333 938

- D1+D2+D3=0.95………………. (13)
- $D_0^* \lambda_0 = m_f^* C_f (T_1 T_f) + D_1^* \lambda_1$

DO*2230.038 = 1*4.17005 (99.5-30) +D1* 2256.816…………………………………(14)

• $D_2^* \lambda_2 = (m_f - D_1)^* c p_1 (T_1 - T_2) + D_1^* \lambda_1$

D2*2292.444=(1-D1)*3.92(99.5-86.4)+ D1* 2256.816………………………….…….(15)

• ghD₃* λ_3 =(m_f-D₁.D₂)*cp₂(T₂-T₃)+D₂* λ_2

D3*2333.938=(1-D1-D2)*3.92 (86.4 70)+D2*2292.444………………………(16)

 \bullet By solving above equations (8) and (9) and (10) and (11) we get:

 $D_{O} = 0.461 \text{Kg/S}$

- $D_1 = 0.327$ Kg/S
- $D_2 = 0.337$ Kg/S
- $D_3 = 0.287$ Kg/S
	- For $1th$ effect; we calculate concentrate of salt

From e.g (5):

$$
xp1 = \frac{1 * 0.03}{(1 - 0.327)} = 0.04
$$

• For $2nd$ effect we calculate concentrate of salt

From e.g. (6):

$$
xp2 = \frac{1 * 0.03}{(1 - 0.327 - 0.337)} = 0.089
$$

The Mass Balance for all the effects can be given as:

$$
\frac{\text{For 1}^{\text{st}}\text{effect:}}{SE = \frac{0.95}{0.461} = 2.06}
$$
\n
$$
A1 = A2 = A3 = \frac{DO \cdot \lambda_0}{(U \cdot \Delta T)}
$$
\n
$$
A = \frac{0.461 \cdot 2230 \cdot 0038}{(2.5 \cdot 10.50)} = 39_{\text{m}^2}
$$

• To calculate no of tubes:

A=л*no of tubes*O. D of tube *length of tube

• Take O.D of tube = 50.8mm and length of tube=6m

$$
\frac{A}{NT = (n \cdot D \cdot L)} = 40.7 \approx 41 \times 100 \times 10^{-39} = 40.7 \approx 41 \times 1000
$$

Basicity Download:

If the base of the chromium solution is prepared with 10 kg of dichromate and the base of 45%, it should go down to 33

The solution

Basal lowering is performed by adding the acid.

Total weight of chromium in solution $= .3.49$ kg.

The base is in the range $(45-33) = 12%$ of the following equation:

 $2Cr(OH)_{3} + 3H2SO4 \rightarrow Cr2(SO4)$ 3 + 2O

104 kg of chromium needs 294 kg of sulfuric acid to lower the basal extent over 100%.

Or

3.49 kg chromium requires a kilogram of sulfuric acid to lower the base in 100% range. Or

3.49 kg, you need 294 x 3.49 of the sulfuric acid to lower the base in the 100% range.

104

Or

3.49 kg, you need 294 x 3.49 of sulfuric acid to lower the base in the 12% range

 $= 100 \times 104$

 $= 1.184$ kg, Sulfuric acid.

Answer = 1.148 kg of concentrated acid.

Hence the basal expense has three things that you should know.

1- Total weight of chrome.

2- Raising or reducing basal.

3- The extent to which the rule is intended to change.

Basicity lift

Example of raising basal from 40% to 50% Chrome solution was prepared with 5 kilograms of sodium dichromate and the Basicity was 40%. What is required to change the base to 50% for this solution?

The solution

5 Kilos of Dichromate = 1.745 Kilometers Chrome

From the chemical equation

 $2Cr10$ (OH) 12 (SO4) $9 + 3Na2CO3 + 6H2O = 5Cr4$ (OH) 6 (SO4) $3 + 3Na2SO4 + 3CO2$ + 3H2O

(40% basic) 50% basic

When you learn that 1040 kg of chromium needs 318 kg of carbonate soda to change the base from 40% to 50% or 1.745 kg of chromium needs 318 x 1.745 kg of carbonate.

 $(318 \times 1.745) \div 100 \text{ kg}$ soda carbon = 0.5335 kg soda carbon.

0.5335 kg soda carbonate changed the basal from 40% to 50%

Noticeable:

In this way the basal can be raised but this method takes a lot of time.

 Photo (A.11) Gravity Separators: (The snail) The snail consists of connections in the form of circular segments.

 Photo (A.12) chromites ore packages for Export