BINDURA UNIVERSITY OF SCIENCE EDUCATION

FACULTY OF SCIENCE EDUCATION

CHEMISTRY DEPARTMENT

PRODUCTION OF RED OXIDE FROM SCRAP METAL

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DISSERTATION SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE BACHELOR OF SCIENCE HONOURS DEGREE IN CHEMISTRY

SEPTEMBER 2022

FACULTY OF SICENCE EDUCATION

DEPARTMENT OF SCIENCE AND MATHEMATICS EDUCATION

Approval form

The undersigned certify that they have read and recommended to the Bindura University of Science Education for acceptance; a research project submitted in partial fulfillment of the requirements for the Bachelor of Science Honors Degree in Chemistry Education

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ACKNOWLEDGEMENT

In the first place I stand to salute the uncnditional support that I received from my family during the course of the research and this can not be all without The mention of my wife Sarirei Dzimureka who pillared with me through. Also the provision of abundant grace from the Almighty who allowed it all. Without my dearest and ever present to assist Professor .Mupa all could have failed but he was there for it all, I thanks you Professor for the part so played

ABSTRACT

In this article the production of red oxide as a pigment used in various areas as a coat to water proof and or paint has been reviewed .The scattered metal wastes and their being a pollutant in the present day 21st century generation remains a major challenge that needs a second consideration. Thus a consolidated investigation on how to make use of scrape metal that Is all over the streets and dump yards such as car centenaries. Scrape metal being the major feed stock adopted due to its abundance as an industrial solid metal wastes (I.S.M.W.)

The main purpose of this review paper Is to zero in on the production of Red Oxide and precisely give on top of the available Information to the production of Red Oxide from scrape metal hence realize the potential with the commercial Red Oxide that Is in the market. In the research work the following were studied hence compromise conditions were used based on the available and experimental outcomes.

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DECLARATION.

I Clever Kudehama registration number B 211921B ,do here stand to declare that all the work in this project Is my own produce and has not been published with any of the Universities . It Is submitted in partial fulfilment of the requirements of the Bachelor of science honors degree (Chemistry) In the faculty of science Education at BINDURA UNIVERSITY OF SCIENCE EDUCATION

Signed……………………………………………….Date…………………………………
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ACRONMYS

Fe	iron
H_2SO_4	sulphuric acid
I.S.W	Industrial solid wastes
KOH	potassium Hydroxide
NH_3	ammonia
NaOH	Sodium Hydroxide

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CHAPTER 1: INTRODUCTION

1.0 Introduction.

This CHAPTER looks at the background to the study ,the statement of the problem , research objectives, purpose of the study , Significance of the study ,delimitation , Limitations and some possible definition of terms.

1.1 Background

The increase in the amount of scrape metal in the residential backyards ,streets and car centenaries at the expense of the potential useful products from the same scrape wastes and its potential to be a health hazard causing tetanus when one gets pricked by a rust metal has greatly triggered the need to choose to produce red oxide from scrape metal as feed stock. This will beside benefiting the producer Is by a fraction going to reduce amount of solid wastes in the environment

This invention relates to the manufacture of red iron oxide products. More particularly, it is concerned with the production of red iron oxide pigments, without a calcination step, by the oxidation of metallic iron, while immersed in a solution containing precipitated iron hydrate

1.2 Problem statement

Most People; n the rural Zimbabwe and even in the urban are patients of tetanus and can not trace the sources since they get wounds from scrape metal during their youthfull ages as they play in the backyards and car cemetry.Besides the cost of buying oil paints for homes ,roofs and even floors Is too high. The need to reduce the solid metal wastes in the homes and car cemetery by re-use of scrape metal to produce other products as Red Oxide . The paints from other sources trigger asthma hence an alternative.

1.3 Significance of study

This intention of this research Is to layout the form foundation for the production of Red Oxide from scrape metal .The production of a relatively low cost pigment remains the goal of this research besides the re-use of scrape metal into other benefiting products. Red Oxide Is best pigment for the domestic use given the high cost of other paints especially the oil paints.

1.4 Justification of study

Red Oxide Is attracting most households as an alternative pigment for their roofs and floors because it Is cheap and reliable .The new development to produce red oxide from scrape metal will reduce solid wastes, create employment and prove the stands of living in People .This being so ,the study aim to embrace the idea locally in Zimbabwe to enjoy the basic advantages in Red oxide.

Red iron oxide is known by many names such as red ochre and bloodstone. Native peoples in the Indian subcontinent and the Americas extensively paid attention to it in geology and used it for decorative purposes and images. For exhibiting body paint, it was also used as a raw material. Hematite is the first choice ore as a source of iron for manufacturing iron and steel. The purest form of hematite is referred to as 'rouge' and it is used to polish glass and plates – capitalising on its abrasive qualities related to its hardness and strength.

A naturally occurring iron oxide ore is thin and scaly and is called micaceous hematite. It is reported that this material is used as a substitute or in the manufacturing of stoves replacing black lead. It also holds immense importance in petroleum and natural gas industries as a lubricant, pivotal in oil drilling (see later). Iron mine tailings are rich in haematite, which is how they can be used to manufacture bricks. Recycling is required because of the high alkalinity of the tailings; thus it can be found easily in recyclable waste streams in large volumes.

One of the choices to give pottery its shiny characteristic, hematite can be used. It has been used as pottery glazing and was mixed with enough water to make it easy to apply. In earlier centuries, tiles and bricks were decorated using haematite and pastes made of hematite.

Anti-corrosion paint is also made by using red iron oxide. Hematite is inert towards ultraviolet rays, making it the perfect anti-corrosive barrier. It can be used in paints and roof tiles to make sure that there is no little to no exposure to ultraviolet rays to the structure underneath. It can also shield from harmful effects of sulphur dioxide, ammonia and other pollutants. Even when making buildings, it can be used with other coarse materials such as cement. Iron tailings can help make unfired bricks due to their characteristics of being strong and durable after being reprocessed – a popular construction method in the developing world. In optimal pressure conditions and adequate water content, iron tailings can form non-fired bricks and other miscellaneous building materials

Red iron oxide pigments are commercially produced by the calcination of copperas and by the calcination of hydrates of iron which have been prepared by chemical precipitation from ferrous or ferric iron containing solutions, or by mixing iron salts with alkaline materials in the dry state followed by calcination of the mixtures. Numerous attempts have been made to produce red iron oxide pigments without a calcination step utilizing the wet process type of operation which has been successfully employed for the production of yellow or brown oxide pigments of the hydrate type. For example, various shades of yellow pigments of hydrated iron oxides had been prepared commercially by precipitation from aqueous solutions using procedures

1.5 Aim

To produce red oxide from scrape metal

1.6 OBJECTIVES

The objectives of this project are to be able to :

- Collect scrape metal suitable for red oxide production.
- Produce red oxide from scrape metal.
- Reduce environmental solid waste .
- Asses the potential employment in red oxide production.
- Give alternative methods of producing red oxide besides the wet method.
- Make money from selling red oxide to local community

1.7 Delimitations

The research Is going to be carried out at BUSE ASTRA campus chemistry laboratory

1.8 Limitations.

The researcher will have some technical Challenges due to limited research skills and most will be consolidated from my supervisor Prof.Mupa.M

CHAPTER 2 LITERATURE REVIEW

2.0. INTRODUCTION

This chapter Is going to investigate the morphology behind Red Oxide .Also give the structure of Red Oxide as a compound before giving an investigation on factors that affect Red Oxide production from scrap metal that include Base added temperature. This will Also lead to investigation of possible colour of pigments from each base treatment .

2.1.The morphology behind Red Oxide

Red oxide (lead oxide) is lead monoxide or litharge (PbO) lead tetra oxide or red lead oxide (Pb3 O4) and black or gray oxide which is a mixture of lead monoxide and metallic lead with a ratio of 70:30 especially that used in lead acid storage battery manufacturing. Litharge is used primarily in the in the manufacturer of various ceramic products. Due to its electrical and electronic properties, litharge is also used in capacitors , Videocon tubes and electro photographic plates and in ferromagnetic and Ferro electric materials. It is also used as an activator in rubber ,a curing agent in elastomers a sulphate removal agent in the production of thioles and in production of lead Chemicals, drying colours,soap and driers for paint including in the production of lead salts used to stabilize plastic like the PVCs.

The major pigment is red oxide Pb3O4, which is used principally in ferrous metal protective paints.

Red iron oxides as the pivot of this research are commonly used as pigments for colouring of construction materials, paints and coatings, plastics, paper, cosmetics and the like. As a pigment it can be spread in the form of a thin layer that will form a solid adherent and cohesive film. The paint is a surface coating fluid material. Simply put a paint is a coating applied to surfaces or substrate to decorate and protect it or perform some other tailor made functions. In addition they may be used as catalysts in chemical reactions such as conversion of ethylbenzene to styrene and as burn rate control agents for solid rocket fuel. For these applications products typically have a small (0.1 micron to 3 micron particle size and preferably consist of pure hematite crystals with minimal content of other iron oxide crystal types.

Most of the processes commonly used to produce red iron oxides employ iron sulphate as starting material. However, iron sulphate feedstock is becoming increasingly difficult and expensive to obtain due to scarcity of titanium dioxide plants based on sulphate technology, reduction of steel pickling operations using sulphuric acid in the country and increased use of ferrous sulphate as a reductive for chromium (VI) in the cement industry. The alternative of dissolving scrap steel in sulphuric acid is becoming increasingly costly as well and necessitates design of reactors to manage the hydrogen liberated.

Preparation of red oxide is based on the reaction litharge with acetic acid bor acetic ions . It can be produced by oxidizing litharge in a reverberatory furnace. This commercial lead can also be produced by wet chemical method, a thermal process in which lead is directly oxidized with air and usually either at low temperature below melting point of lead and or moderate temperature between the melting point of lead and lead monoxide.

The low temperature preparation is achieved by tumbling slugs of metallic lead in a ball mill equipped with an air flow for oxygen as well as a coolant. Cooling is to keep the temperature from oxidation reaction and mechanical heat from tumbling.

In addition, environmental concerns are related to the red iron oxides production processes based on iron sulphate. During annealing of iron sulphate, sulphur gases are released which must be collected and converted back to sulphuric acid in a sulphuric acid plant while not reacted iron sulphate in the product is typically neutralized to give a gypsum waste stream. In precipitation processes a salt solution is generated which must be treated before release to the environment, typically by evaporation to sodium or ammonium sulphate. Moreover, red iron oxides obtained from sulphate precipitation processes usually contain significant quantities of goethite which reduces the brightness and colouring strength of the hematite pigment.

In contrast to ferrous sulphate solutions, ferrous chloride solutions are more readily available, for example, from pickling of steel or beneficiation of ilmenite ores by acid leaching processes.

Typically, ferrous chloride pickling wastes have been disposed by roasting to recycle the hydrochloric acid value while generating a very low value iron oxide by-product. Alternately, ferrous chloride pickling wastes are neutralized and disposed in a landfill. However, the ferrous chloride by-products contain a significant level of impurities such as manganese and zinc, which can have negative effects on pigment production processes.

A process for production of red iron oxide pigments from ferrous chloride solutions with the added advantage of calcium chloride recovery . The process involves neutralization of the ferrous chloride with calcium hydroxide followed by oxidation over 4 to 6 hours, separation of the calcium chloride solution for recovery, washing, drying and calcining the product iron oxide at 750° C. to 800° C. to achieve the target iron oxide.

A process for precipitation of red iron oxides without a calcination step. Metallic iron is treated with oxygen containing gases in an aqueous iron (II) salt solution containing seeds of iron oxide or iron oxide hydroxide, wherein the seeds are prepared from precipitation of iron (II) ions by addition of alkali or alkaline earth hydroxides or carbonates. Both ferrous sulphate and ferrous chloride are suitable

It is an object of the present invention to provide a process for producing high quality red iron oxides from a ferrous chloride feedstock.

There are significant differences between growth of red oxides in chloride and sulphate systems. In the sulphate process the seed is typically a mixture of various iron oxide species including but not limited to one or more of hematite, goethite, lepidocrocite and/or magnetite. When grown at normal conditions in the presence of metallic iron at elevated temperatures all of these species typically convert to small particle hematite. However, in the case of chloride feedstock the goethite seeds will not transform to hematite but persist as goethite throughout the growth reaction with a significant negative effect on the red colour quality.

Thus, it is a further object of the present invention to provide a process for producing red iron oxides being substantially free of goethite.

It is a further object of the present invention to provide a process for producing red iron oxides that utilizes a ferrous chloride by-product as a ferrous chloride feedstock

In one embodiment of the invention, a process for producing red iron oxide is provided, comprising the steps of providing a ferrous chloride feedstock, precipitating high surface area lepidocrocite seeds having a BET surface area of greater than about 175 m2/g by mixing the ferrous chloride feedstock with an alkali, oxidizing the obtained mixture, and growing the lepidocrocite seeds, whereby the lepidocrocite converts into red iron oxide.

In another embodiment of the invention, high surface area lepidocrocite seeds having a BET surface area of greater than about 175 m2/g are provided.

2.2 Fe_2O_3

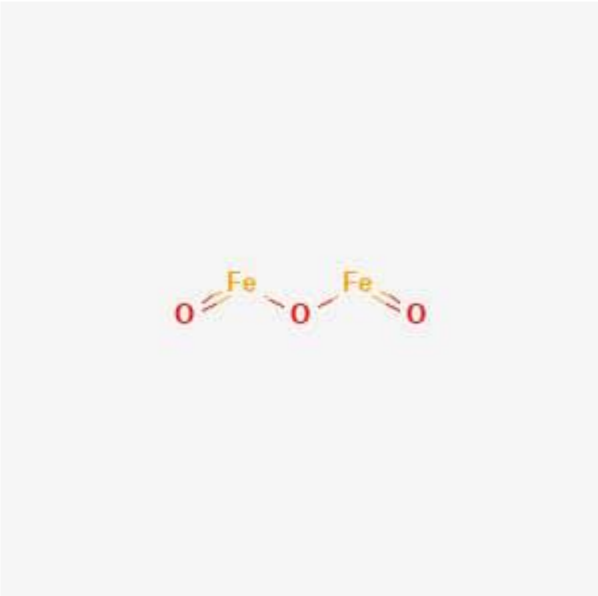

Figure2: 1 The chemical structure of Red Oxide Is as below

2.3 Factors affecting production of Red Oxide

2.3.1 Base used

The addition of bases result in visible colours that are suitable to be used as dyes, food colouring, and indicators of iron contaminations. Bases such as sodium hydroxide (NaOH), ammonia (NH_3) and potasium hydroxide where used based on availability.

2.3.2 Tempature

The thermal treatment of the pigments from each produced some different results that Showed temperature affects the production of Red Oxide from scrape metal

2.4.Types of Red Oxide (colours produced)

Black, Red, Green and Grey

Table 2: 1 Below describe the types of Red Oxide colours that obtains from each Base treatment

Test tube	Base added	Observation
1	$NaCO_3$	Black precipitate
2	NaOH	Grey precipitate
3	NH_3	Red precipitate
4	KOH	Green precipitate

CHAPTER 3 METHODOLOGY

3.0 INTRODUCTION

3.1 Material

The following material and chemicals were used to carry out the research project;

- Iron scrap
- Sulphuric Acid
- Sodium carbonate
- Sodium Hydroxide
- Ammonia
- Potassium Hydroxide

APPARATUS used

The following apparatus were used; Two necked flask, Dropping funnel/ separating funnel, Condenser, Magnetic stirrer, Filter paper, Vacuum filtrator, Conical flask, Measuring cylinder,

Figure 3: 1a Spatula, Scale and a Heater.

Figure 3: 2 Heat source

3.2 PROCEDURE /METHODOLOGY

One mole(56g) of scrap iron fillings was weighed on a scale and poured into the two necked flask

The condenser was connected into the two necked flask with the water in and out tubes connected.

The flask was mounted on the reflux

The separating funnel was connected with the valve closed

Sulphuric Acid was loaded into the separating funnel

The Acid was added drop wise into the flask

The heater was switched on to 30 degrees Celsius

The mixture was left for 3 hours to allow complete reaction

The mixture was filtered on a vacuum filter

The light green filtrate was concentrated by evaporation until it was bottle green

The filtrate was left in the fridge over night at 4 degrees celcius

On day two the filtrate was poured into four test tubes

The reaction equations

1.controlled rusting of iron:

$Fe + H_2SO_4 \rightarrow FeSO_4 + H_2$

2.$FeSO_4 + NaOH \rightarrow Na_2SO_4 + Fe(OH)_2$

Ferric Oxide Hydroxide, $Fe_2O_3 \cdot H_2O$ (synthetic goethite)

3.The growth phase: the seed solution was fed reactor with scrape iron. Circulation with air causes iron to pass into solution at The same time that iron Oxide Is depositing out of solution on to the growth nuclei.

The product was obtained by filtering The solution by vacuum filtration and washing The recovered precipitate. The purification was carried out at an acidic pH value a pH from about 3 to 5. This produced a bluewish green solution.

In exemplary embodiments of the invention, the growing of the lepidocrocite seeds is accomplished by adding further ferrous chloride feedstock and alkali to the obtained mixture comprising the precipitated high surface lepidocrocite seeds. For example, the ferrous chloride feedstock can be used undiluted, having an ferrous iron concentration from about 5 to 23%, preferably about 15%. The alkali may be one of those described above. The addition may be done simultaneously or sequentially in any order. During the growth step, the lepidocrocite converts into red iron oxide. The growing of the lepidocrocite seeds can be carried out at temperatures equal or above 80° C., such as from about 80 to 110° C., such as from about 85 to 95° C., preferably from about 90 to 95° C., at a suitable pH of the mixture, depending on the type, amount and ratio of alkali and ferrous chloride feedstock added. In exemplary embodiments, the growing of the seeds can be carried out at an overall acidic pH, e.g. a pH value from about 3.5 to 5.5.

3.3.Determination of Red Oxide yield

The percentage yield Is calculated using the formula below,

Actual mass obtained / calculated mass x100% then the percentage purity Is calculated using the formula, mass of pure product /mass of impure product x 100%

CHAPTER 4 RESULTS AND DISCUSSION

4.0 INTRODUCTION

This chapter Is going to concentrate on the results of the experimental work conducted during the research process on the production of Red Oxide using scrape metal .The results were obtained and complied during the production of Red Oxide . The thrust of the research work was to investigate and analyse the applicability of producing Red Oxide using scrape metal and then the factors that affect the production of Red Oxide to include Base used (pH) and temperature after thermal treatment

4.1.Determination of Red Oxide yield

The percentage yield was calculated using the formula actual mass of Red Oxide sample/ calculated mass from experimental work x100%

4.2 Pigment analysis and the selected factors as shown below

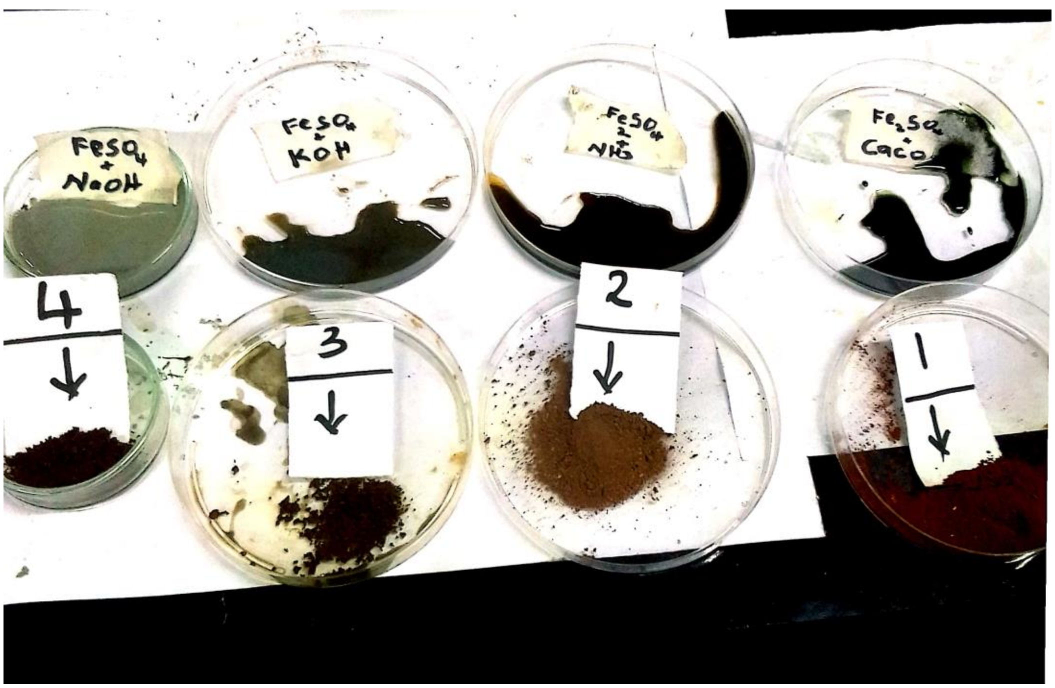

Figure 4: 1 Pigment analysis and the selected factors as shown on fig 2 below

Figure 4: 2 Mixture of iron-base mixture

4.3 Effects of base used

Taking into account all the discussions ,optimal parameters of Red Oxide pigment produced from scrap metal were 12:39 iron to sulphuric acid molar ratio and sodium hydrosulphite dehydrate as Red Oxide reagent. The characteristic property of Red Oxide pigment prepared under optimal parameters were compared

From the selected bases ,NH3 produced The deep Red Oxide that Is mostly used as a pigment in many homes and industries in Zimbabwe

4.4 Effects of temperature

After a thermal treatment of all samples from each test tube The colour that obtained from each was different as on the fig 4

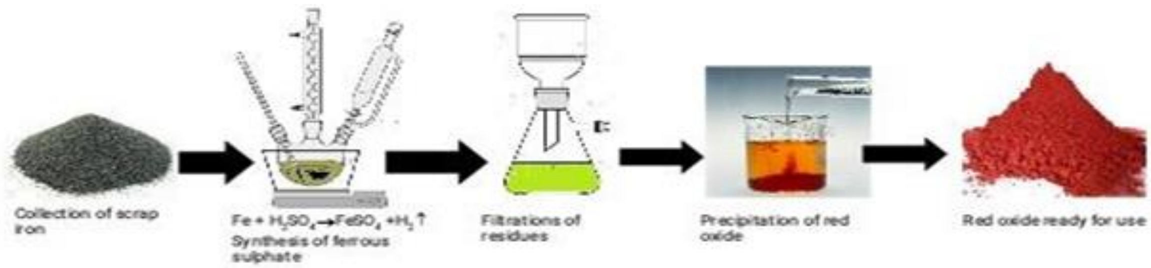

Figure 4: 3 Illustration of synthesis steps for red oxide

Figure 4: 4 Synthesis step for preparation of red oxide through thermal decomposition of ferrous sulphate

CHAPTER 5

5.0 Conclusion

The objectives of the research were met as Planned by the researcher. It can be summerised as a conclusion that Red Oxide from scrape metal with 89% yield was achieved and this was due to effects of impurities and use of compromise conditions .The chemical redox reactions that occurred were Also under compromise conditions. This Also affected the purity of the Red Oxide obtained since the material and chemicals used were not pressure washed.

5.1 Recommendations

It Is recommended to further studies on the methods of producing Red Oxide using other feed stocks and or even same scrape metal .It Is Also recommended to carry out a pilot study on the advantages of Red Oxide claimed by this paper

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