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Production of Ferrous Sulphate Heptahydrate from Waste Iron Nails

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ABSTRACT

This research aimed to produce ferrous sulphate heptahydrate crystals from waste iron nails collected from a building construction site in Nigeria using simple laboratory methods to emphasize the importance of converting wastes to useful products. A 20% tetraoxosulphate (VI) acid was used to extract pure iron from the iron nails, resulting in a solution of ferrous sulphate that was then crystallized from the solution. The percentage yield of the ferrous sulphate crystals from 10 g of iron nails used in the experiment was 62%, indicating impurities in the nails. The water of crystallization was calculated to be 41%. A precipitate of dirty green colour was observed when sodium hydroxide solution was added to a sample of the freshly prepared ferrous sulphate solution which confirmed the presence of ferrous sulphate.

Keywords: Crystallization, Ferrous Sulphate, Hydrated, Iron, Waste.

INTRODUCTION

Nigeria is a developing nation and with a population of over 230 million people [1], shelter is of basic concern. Building constructions are daily going on in most towns and cities with a number of wastes generated. These include metal wastes such as iron nails, copper wires, pieces of corrugated iron and aluminium roofing sheets; plastic wastes; glass wastes; etc. However, these waste materials are not fully utilized and a good portion of them end up being discarded to the environment where they constitute nuisance [2, 3]. Rather than discarding these wastes, they can be converted into useful products [4, 5]

Ferrous sulphate is a useful product that can be produced from metal waste. Ferrous sulphate also known as Iron(II) Sulphate, encompasses a variety of salts with the formula $FeSO_4 \cdot xH_2O$. These salts in most cases are found as the heptahydrate with seven waters of crystallization ($FeSO_4.7H_2O$). However, other values of x are also known. Ferrous sulphate heptahydrate is usually formed from the reaction of iron with Tetraoxosulphate (vi) acid. This reaction is a displacement reaction where the iron atom replaces the hydrogen atom in the acid thereby expelling hydrogen gas. The resulting salt when crystallized out is ferrous sulphate heptahydrate. The chemical reaction is:

$Fe(s)nails + H_2SO_4(aq) \rightarrow FeSO_4(aq) + H_2(g)$ …………………… (1)

Upon crystallization

$FeSO_4 + 7H_2O \rightarrow FeSO_4.7H_2O$ (crystals) ……………………... (2)

Ferrous sulphate heptahydrate is widely used in medicine and pharmaceuticals, agriculture, textile, water treatment, ink production and as raw material for complementary industries [6-8].

Commercially, ferrous sulphate heptahydrate is produced primarily as a byproduct of steel pickling with tetraoxosulphate (VI) acid and in the production of titanium (IV) oxide. It can also be produced through the oxidation of pyrite and by the displacement of metals less reactive than iron from solutions of their sulfates [7]. Other than these commercial scale methods of production, this research was set out to highlight an alternative way in which locally available materials could be used to produce ferrous sulphate that would compete favourably with the commercial ones.

The objective of this work was to demonstrate that daily waste materials can be converted into useful products. This was achieved by synthesizing ferrous sulphate crystals from waste iron nails collected from a construction site in Abraka.

MATERIALS AND METHODS

The chemicals used for the synthesis and analysis were of analytical grade and Sigma Aldrich branded as purchased. They include, concentrated Tetraoxosulphate(VI) acid, H_2SO_4 (94%) and Sodium hydroxide, NaOH (1.4 M).

Sample Collection and Preparation

Wastes iron nails were sourced from a building construction site at Abraka town in Delta State of Nigeria. The nails were washed thoroughly with warm water to remove any dirt, dried and weighed.

Production of Ferrous Sulphate Crystals

Exactly 20% of tetraoxosulphate(VI) acid was first prepared using distilled water. Approximately 50 ml of the acid prepared was then measured into a beaker. Approximately 10 g of nail pieces was weighed and carefully placed inside the beaker of the 20% acid and left to react. The reaction went on until all the pieces of nail had been dissolved into the solution and hydrogen gas had stopped emitting. Then, the solution was further heated to concentrate it, then filtered and the filtrate collected and kept in a water bath and left all night to allow the ferrous sulphate crystals crystallize out. The crystals formed were filtered out and further recrystallized using water. Thereafter, the crystals were finally filtered, dried and weighed [9].

The equation of the reaction is given as;

$Fe(s)nails + H_2SO_4(aq) \rightarrow FeSO_4(aq) + H_2(g)$ ……………………………..… (3)

$FeSO_4(aq) + 7H_2O \rightarrow FeSO_4.7H_2O$ (s) (on crystallization) ……………..…. (4)

Determination of Percentage Yield

The percentage yield was deduced from the relation between the actual mass of ferrous sulphate obtained and the theoretical yield [10]

Percent yield = Actual mass of ferrous sulphate obtained/Theoretical yield of ferrous sulphate **x** 100%

Where;

Theoretical Yield = (Mass of iron used **x** 1Mole of iron / Atomic Mass of iron) **x** (Moles of ferrous sulphate Produced / moles of iron Used) **x** (Molecular Weight of ferrous sulphate heptahydrate / 1Mole of ferrous sulphate.)

Test for Ferrous Sulphate

To test for ferrous sulphate, 5 ml of the freshly prepared ferrous sulphate solution was put in a test tube. A dropping pipette was then used to add sodium hydroxide solution to it until a colour change was observed [10].

Determination of Water of Crystallization of Synthesized Ferrous Sulphate Heptahydrate

Using a weighing balance (Mettler Toledo), 3.0 g of the ferrous sulphate crystals was measured in a dish and dried at 90 ^{0}C to a constant weight in an oven. The difference in weight was noted [10].

$FeSO_4.7H_2O (s) \xrightarrow{\Delta} FeSO_4(s) + 7H_2O(g)$ ……………………………… (5)

RESULTS AND DISCUSSION

Yield of Ferrous Sulphate from Waste Iron Nails

The yield of ferrous sulphate crystals in percentage is shown in Table 1.

Table 1: Yield of Ferrous Sulphate from Waste Iron Nails

Mass of iron Used	10.00 g
Theoretical Yield of Ferrous Sulphate	49.82 g
Mass of Ferrous Sulphate Produced	31.00 g
Percentage Yield of Ferrous Sulphate	62.00%

Determination of Water of Crystallization

The result obtained for the determination of water of crystallization is given in Table 2.

Table 2: Determination of Water of Crystallization of Ferrous Sulphate Heptahydrate

Initial mass of $FeSO_4$ crystals	3.00 g
Final Mass of $FeSO_4$	1.78 g
Water of crystallization	41.0%

Test for Ferrous Sulphate

The test observations made for ferrous sulphate are as tabulated in Table 3.

Table 3: Test for Ferrous Sulphate

Activity	Colour Observed
Freshly Prepared $FeSO_4$	Bluish green
Addition of NaOH to $FeSO_4$	Dirty green
Standing after 30 min.	Brown

Table 1 showed the yield of the synthesized Ferrous Sulphate heptahydrate. It indicated that the weight of the crystals obtained from 10 g of the waste iron nails used was 31.00 g. Therefore, the percentage yield of the produced salt was calculated to be 62.00%.

The difference in the theoretical yield of the ferrous sulphate and the actual yield of ferrous sulphate obtained could be attributed to impurities that may be present in the composition of the nails [11].

Table 2 showed the value of Water of Crystallization of the ferrous sulphate crystals synthesized. The presence of water of crystallization was indicated by the difference in mass of the crystals before and after dryness to a constant weight. The initial mass of the ferrous sulphate crystals weighed was 3.00 g and after dryness to a constant weight, the mass became 1.78 g. The difference in weight, 1.22 g gave the water of crystallization which was expressed as 41%.

Table 3 showed a simple laboratory identification test for ferrous sulphate. When sodium hydroxide solution is added to a freshly prepared solution of ferrous sulphate, a dirty green precipitate of ferrous hydroxide will be formed [12]:

$FeSO_4$ (aq) + 2NaOH (aq) → $Fe(OH)_2$ (s) + Na_2SO_4(aq) …………………………… (6)

This was also observed when sodium hydroxide solution was pipetted drop-wise into a test tube of the freshly prepared ferrous sulphate solution before crystallization. The bluish green colour of the solution gradually turned to a dirty green insoluble precipitate of ferrous hydroxide, therefore confirming that the solution was ferrous sulphate. It was also observed that the precipitate changed from dirty green to brown on standing. This showed the instability of the precipitate which further oxidizes to form the brown Ferric hydroxide (iron (III) hydroxide;

$Fe(OH)_2$ → $Fe(OH)_3$ (oxidation) ……………………… …………(7)

CONCLUSION

Building sites are potential sources of iron waste which can serve as secondary raw materials for producing useful products. This work demonstrated that ferrous sulphate crystals can be synthesized from waste iron nails obtained from a building construction site, using simple laboratory procedures that can be conveniently carried out. It can be determined that useful products can be derived from waste items.

Although the research was limited to waste iron nails, there are several other waste materials that can be found in building construction sites. It is recommended that these other waste materials be studied with the aim of converting them to useful products.

The applications of ferrous sulphate heptahydrate apply to several areas that require different specifications and levels of purity. Future works will therefore focus on purifying ferrous sulphate heptahydrate to meet target applications.

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