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Quantitative Production of High Concentration Poly-Ferric Magnesium Sulfate Coagulant Through Ferrous Sulfate Oxidation

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Abstract: Poly-ferric magnesium sulfate coagulant as another water purifying agent besides poly-aluminum multi-salt can be quantitatively produced through ferrous sulfate oxidation with high product concentration. This article starts with the selection of raw material, then elaborates process flow from a small-scale lab simulate experiment to a large-scale production of this high concentration water purifying agent. According to the corporate technical standard of this product, chemical compositions of the primary material (ferrous sulfate heptahydrate) and auxiliary materials (alkalizing agent, oxidizer, etc.), and the alkalinity of the product (B=8%), this article provides calculation of the raw materials' input and output for producing 1 ton of the product liquid. The reaction is carried out step-by-step in a heating reactor equipped with acidic mist re-flux tower and filtration devices. The first step: ferrous sulfate oxidation by sodium chlorate; the second step: polymerization, precipitation, and filtration separation; the third step: arsenic and heavy metal check and precipitate separation by flocculating precipitant and metal chelating precipitant; last, use the laboratory test results to back calculating alkalinity (B%), coagulation strength (C%), and molecular formula of the final product liquid. This article not only rationalizes the feasibility with chemical/physical mechanisms this technique, formulation, production method, but also guides through the back calculation to provide data of the properties as a documentation of the certificate of analysis for the product.

Keywords: Ferrous Sulfate, Sodium Chlorate, Magnesite Powder, Coagulation Strength, Arsenic Flocculating Precipitant, Heavy Metal Chelating Precipitant, Poly-Ferric Magnesium Sulfate Coagulant

1. Introduction

To manufacture an ideal water purifying agent, a higher concentration (or higher yield), a wider coagulation range, and a lower cost are the considerations for a manufacturer to plan with. Poly-ferric magnesium sulfate coagulant as another water purifying agent besides poly-aluminum multi-salt can be quantitatively produced through ferrous sulfate oxidation with high product concentration. This article starts with the selection of raw material, then elaborates process flow from a small-scale lab simulate experiment to a large-scale production of this high concentration water purifying agent.

According to the corporate technical standard of this product, chemical compositions of the primary material (ferrous sulfate heptahydrate) and auxiliary materials (alkalizing agent/magnesite powder, oxidizer/sodium chlorate and sulfuric acid), and the alkalinity of the product (B=8%), this article provides calculation of the raw materials' input and output for producing 1 ton of the product liquid. The reaction is carried out step-by-step in a heating reactor equipped with acidic mist re-flux tower and filtration devices. The first step: ferrous sulfate oxidation by sodium chlorate; the second step: polymerization, precipitation, and filtration separation; the third step: arsenic and heavy metal check and precipitate separation by flocculating precipitant and metal chelating precipitant; last, use the laboratory test results to back calculating alkalinity (B%), coagulation strength (C%), and molecular formula of the final product liquid.

This water purifying coagulant contains a major salt - ferric sulfate. The primary source for ferric sulfate is ferrous sulfate - a manufacture's byproduct that we can find from a titanium dioxide production plant. This byproduct contains abundant ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$). There is a small amount of free sulfuric acid in this byproduct. Another source of the ferrous sulfate is the waste solution from a steel manufacture that has high concentration of ferrous sulfate. The chemical reactions under the given conditions and meaning of the alkalinity are outlined in this section.

1.1. Ferrous Sulfate (Fe^{2+}) Oxidation to Form Ferric Sulfate (Fe^{3+})

The two ordinary processes for oxidizing ferrous sulfate under acidic condition are (1) sodium chlorate oxidation; (2) nitrogen nitrate oxidation with spray pump recycling process. Based on the process (1), ferrous ion (Fe^{2+}) must be oxidized to Ferric ion (Fe^{3+}) so that the coagulation effect for water purification will be more efficient. The pH value for Fe^{2+} ion's precipitation starting point is 6.5 while pH value for Fe^{3+}ion's precipitation starting point is pH=1.6 [1]. The lower pH value will give Fe^{3+} ion wider range for coagulation regarding the impurity removal in water.

1.2. Ferric Sulfate Alkalization and Its Mathematical and Physical Meaning

1) The function and selection of the alkalization agents:

The alkalizing agent is mainly used to adjust the alkalinity (B%) and stabilize the reaction liquid as an acid (H^+)-base (OH^-) buffer. It relieves the impact of acid or alkali on the reaction liquid. It also prevents the reaction liquid from forming of hydroxide precipitate.

Basically, the alkalizing agents are activated bauxite powder [2], synthetic calcium aluminate powder [3], pyrite slag [4], and calcium magnesium carbonate ore powder, etc.

2) Ferric sulfate alkalization:

Ferrous sulfate will be oxidized in acidic solution and form the main ingredient - ferric sulfate heptahydrate complex. The ferric sulfate will go through acid dissolution-hydrolysis-basification, so the adjacent two basic groups will form a network complex [5-8] through bridging polymerization.

3) The chemical and mathematical meaning of alkalinity:

When the primary material, ferric oxide, reacts with certain amount of alkalizing agent (an auxiliary material) it will go through acid dissolution-hydrolysis-basification reactions. The alkalinity (B%) measures the degree of the alkalization or indicates the strength of the bridging-polymerization inside molecules. Alkalinity (B%) can be mathematically expressed as the ratio of the Molar concentration $[OH_t^-]$ of the base group (OH_t^-) of the reaction liquid to the total Molar concentration $[\sum M_i^{n+}]$ of all weak alkali metal ions ($\sum M_i^{n+}$) that participate. The alkalinity can be expressed as:

$$B\%=[OH_t^-]/[\sum M_i^{n+}].$$

4) The chemical and physical meaning of Alkalinity:

(1) The value of the alkalinity controls the amount of acid that to be input into the reaction liquid. The higher the alkalinity the less amount of acid in need. (2) The product must possess certain degree of alkalinity so that it can eliminate free acid in the reaction liquid and ensure the right condition of the later process. It also ensures the safe storage and transportation. Furthermore, it can build alkalizing-bridge polymerizing as mesh complex [5-8]. The physical meaning of the alkalinity is that the forming of the mesh complex with a center ion that possesses the properties of absorbing and capture the negative ion of the turbidity colloid. This is so called coagulation or flocculation [8, 9]. The strength of coagulation depends on the ratio of high valence cations (Al^{3+}, Fe^{3+}, Ti^{4+}) to low valence cations (Ca^{2+}, Mg^{2+}). The higher content of high valence cation in the total cations, the stronger the coagulation, conversely, coagulation is reduced. Therefore, the coagulation strength (C%) can be expressed as the ratio of total molarity of alkalized high valence cations to the sum of molarity of all high and low valence cations:

$$C\%=[\sum M_i^{n\geq 3+}]/[\sum M_i^{n\geq 2+}]$$

The coagulation strength (C%) can be graded as:

Grade I: $C\geq 95\%$ (strongest)

Grade II: $90\%\leq C<95\%$ (stronger)

Grade III: $80\%\leq C<90\%$ (strong)

This article not only rationalizes the feasibility with chemical/physical mechanisms, technique, formulation, production method, but also guides through the back calculation to provide data of the properties as a documentation of the certificate of analysis for the product.

This process of poly-ferric magnesium sulfate coagulant can be quantitatively produced with high product concentration and the product has a wide range of coagulation from the pH as low as 1.6. The reuse of the by-product as a primary material reduces material cost and relieves the burden of pollution treatment.

2. Formulation of Corporate Technical Standard and Preparation of High Concentration Reaction Liquid

2.1. Establish a Corporate Technical Standard

According to Yin and Zhang, the mass of ferric oxide is 160g per mol and the mass fraction concentration in 1M acid solution is 16%, which will be converted into 10%

(=16%/16%x10%) when pH=0 based on the curve of the pH vs. w% (the Fe_2O_3 dissolution rate). From the chart, when pH=0.4, the ferric oxide's dissolution rate is 9.2% [1, 5]. The actual Fe_2O_3 dissolution rate is 9.2%x16%/10%=14.72%.

This dissolution rate can be set as the content of ferric oxide in poly-ferric magnesium sulfate. In auxiliary materials (alkalizing agent + ferric sulfate), the total dissolution rate of other metal oxide, such as aluminum, titanium, calcium, magnesium equals 10%-9.2%=0.8%. The actual dissolution rate of metal oxide from auxiliary materials is 0.8%x16%/10%=1.28%. Thus, the alkalinity of the reaction liquid can be calculated: B%=0.4x10%x2=8% [5].

2.2. Formulation of the High Concentration Reaction Liquid

The amount of ferrous sulfate ($FeSO_4$) determines the degree of the increasing in the concentration of the production reaction solution (the increase multiple). The increase multiple equals actual amount of ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$). If the purity of ferrous sulfate heptahydrate in the primary material is 94%, ferric oxide's content is 27.1% (see section 3.1), that is 1.69 times increases from 16.7% to 27.1% (27.1%/16.0%=1.69) compared with ferric sulfate as the primary material. The ferric oxide dissolution rate from $FeSO_4 \cdot 7H_2O$ is 24.88% (=14.72%×1.69).

Likewise, the amount of other metal oxides (aluminum, titanium, calcium, and magnesium) from auxiliary material (alkalinity agent + ferrous sulfate) will also increase by 1.69 times. For all other metal oxides, the dissolution rate equals 1.28%x1.69=2.16%. Increasing the same multiple times for the amounts of primary materials and auxiliary materials, it not just increases the concentration of oxides, and it also fixes the ratio of the primary material to all materials (primary+auxiliary). It maintains the dissolution rate of primary material, in the meantime, it saves energy that required for product concentration during processing.

2.3. Treatment of Arsenic and Heavy Metals from Reaction Liquid

The arsenic in primary/auxiliary materials dissolve in acid and forms arsenate and arsenite in the reaction solution. The arsenic salts can be identified and removed by using arsenic flocculating precipitant [10, 11]. Other heavy metals dissolved in the solution can be captured and precipitated by using of the heavy metal chelating precipitants [12, 13]. The chelated precipitates of heavy metals are rather stable and will not dissociated at high temperature (within 250°C). Therefore, the heavy metal chelate sediment does not cause secondary pollution to soil/earth. Since the arsenic and heavy metal precipitates have small quantity, they can be accumulated and reclaimed for treatment in a later time. After arsenic and heavy metal treatment, the content of arsenic and heavy metals in the final product will be greatly decreased by $10^{-4} – 10^{-5}$ magnitude.

3. The Primary Material and Auxiliary Materials

Before every batch of production, the manufacture will first work with a small-scale production (1kg) in the laboratory. The small-scale experiment will use 1 Kg formulation. The large-scale batch production will use one ton formulation as a guideline. The small-scale production try-out usually set up reaction liquid (500g – 800g) in a flat-bottom four-neck flask [i] that uses an electric mantle for heating and a size-2 sand core glass plate suction funnel for filtration. The small-scale operation is a simulation of the large-scale production operation.

3.1. The Primary Materials (Fe_2O_3)

The content of ferrous sulfate [14]: calculate percentage of Fe_2O_3 in ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$, purity 94%): Fe_2O_3=94%x80/278=27.1%.

3.2. Alkalizing Agent

(1) Magnesite powder: the magnesite powder is the magnesite ore that crushed and grinded down to 100mesh in particle size. Operator uses 0.149mm (100 mesh) sieve to filter the grinded powder and collect 95% of the powders that passing through 0.149mm sieve.
(2) The chemical ingredients of the Hanyuan's magnesite powder [15]: magnesium oxide (MgO)/43%, calcium oxide/4.76%. Trace iron and manganese calculated as Fe_2O_3 is about 1%, silicone dioxide/1.71%.

3.3. Precipitant

(1) The arsenic flocculating precipitant/PAM+CTS 1% solution [10, 11].
(2) The heavy metal chelating precipitant/TMT-18 2% solution [12, 13].

3.4. Concentrated Sulfuric Acid/Oxidizer

Concentrated sulfuric acid 98%; sodium chlorate: contain $NaClO_3$ 82%.

4. Formulation of the 1 Ton Reaction Liquid: Calculation Basis and Results

According to the technical standard of poly-ferric magnesium sulfate coagulant production, and the chemical contents of primary material (ferrous sulfate heptahydrate) and the auxiliary materials (HanYuan magnesium ore powder and sulfuric acid), and product alkalinity (B%), the formulation calculation for producing 1 ton of production is listed below [16]:

4.1. Material Input Amount for Reaction Liquid

1) Sulfuric acid/178(kg/98%); 2) Ferrous sulfate

heptahydrate ($FeSO_4 \cdot 7H_2O$)/965(kg): contain Fe_2O_3/261(kg/27.1 equivalent sulfuric acid from the sulfuric group in $FeSO_4 \cdot 7H_2O$: H_2SO_4/320(kg); 3) Sodium chlorate/70 (kg/82%): dissolve this amount of sodium chlorate in 280 (kg) water to obtain 20%, 350(kg) chlorate solution; 4) Magnesite powder/48 (kg/49.14%): contain MgO/21 (kg), CaO/2 (kg/convert to calcium sulfate 5kg), contain Fe_2O_3/1 (kg); silicone calcium precipitate sediment/6 (kg).

4.2. The Yield of the Reaction Liquid

1) The total weight of the reaction liquid is 1510 kg (=147+965+350+21). The total amount of reaction liquid is controlled within the range of 1510 ±50 (kg). 2) The actual amount of various materials produced in the production liquid are subject to the actual results from lab measurement.

5. Quantitative Oxidation of Ferrous Sulfate to Produce High Concentration Poly-Ferric Magnesium Sulfate

5.1. The Manufacturing Process

Ferrous sulfate oxidation and poly-ferric magnesium sulfate coagulant's manufacturing process. (Figure 1).

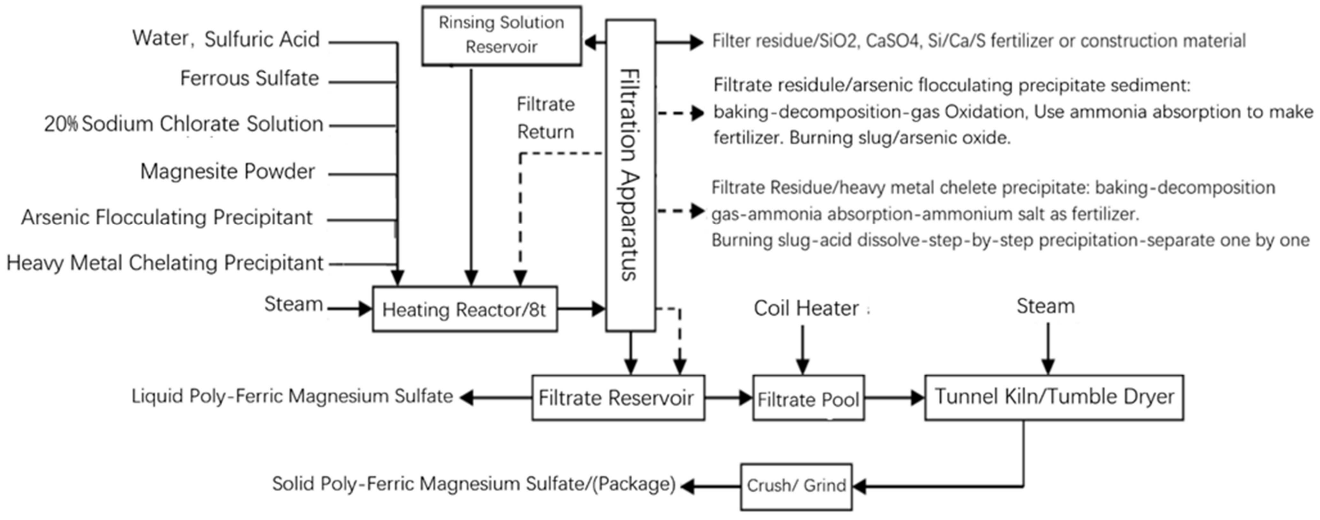

Figure 1: The Oxidation of Ferrous Sulfate and Process Flow for Production of Poly-Ferric Magnesium Sulfate Coagulant

Figure 1. *The Oxidation of Ferrous Sulfate and Process Flow for Production of Poly-Ferric Magnesium Sulfate Coagulant.*

5.2. The Process Operation

The process takes place step-by-step in a combination of heating reactor or a reaction pool that is sealed with arc shape caps and equipped with acidic mist re-flux tower, and filtration apparatus and so on. The process flow chart is shown in the figure above (Figure 1).

Step 1: Oxidation

First, to an 8-ton reactor, add 5x174(kg/98%) sulfuric acid with electric stirrer turned on, then add 5x965 (kg/94%) ferrous sulfate heptahydrate. Next, to the above solution, control the reaction temperature at ≤80°C, slowly add 5x350 (kg/20%) sodium chlorate solution [(preparation: 5x70 kg sodium chlorate dissolved in to 5x280kg water to obtain 20% of 5x350kg solution)]. Control the addition speed slow enough to avoid popping sound. After the completion of ferrous ion oxidized to ferric ion, withdraw some reaction sample from the addition neck of the reactor and check the residue of ferrous ion[ii]. If there is no ferrous ion left, the reaction liquid is completely converted to ferric sulfate solution.

Step 2: Polymerization + precipitation/filtration separation

Polymerization + precipitation: continue stir the above ferric sulfate solution, add 5x48 (kg/43.38%) magnesite powder to the reactor that contains ferric sulfate solution from the first step. With closing of the reactor, the reaction heat will raise up the temperature of the reaction liquid, and this helps the reaction liquid to proceed an acid dissolution-hydrolysis-slight alkalization-bridged polymerization [5-8]. When the reaction temperature reaches to the highest point, it will drop gradually and becomes stabilized. After the temperature of the reaction liquid becomes stabilized for 0.5 hours without change, it indicates the formation of polymeric basic ferric magnesium sulfate solution, silicone oxide, and calcium sulfate, etc. Let the reactor cool down to room temperature, check the pH value. The reaction liquid is qualified if the liquid's pH value is between 0.3~0.5.

Filtration/Separation: return the filtrate liquid back to reactor for next process step; rinse the filter residue with water 2~3 times. Forward the 1st and 2nd rinsing solution to filtrate reservoir and recycle the 3rd rinse solution to rinsing solution reservoir for re-usage. The filtrated residue can be used as construction material or silicone calcium sulfur fertilizer [17-19].

Step 3: Arsenic flocculating precipitation analysis and treatment

Refer to the second step of the section 2.4.2 in the literature [20].

Step 4: Heavy metal chelating precipitation analysis and treatment

Refer to the fourth step of the section 2.4.2 in the literature [20].

6. Calculation the Product Alkalinity and Molecular Formula from the Production Test Results

Measure the production liquid to obtain pH value, density, color, and analysis the production liquid chemical contents [16], then use these data to calculate the alkalinity, coagulation strength, and molecular formula [21] for characterization of the product liquid.

6.1. Conversion of the Moles (i) from Production Tested Weight Percentage Concentration w%

6.1.1. The Basic Molecular Formula

$$\prod M_i=[Fe_y(OH)_{3yB}(SO_4)_{(3y-3yB)/2}Mg_k(OH)_{2kB}(SO_4)_{(2k-2kB)/2}\cdot wH_2O]_{m=1} \quad (1)$$

6.1.2. The Test Results

The weight percent concentration (w%) of the basic metal ion (M^{n+}) of the final product liquid:

Fe_2O_3%=17.5, MgO%=1.4.

the weight percent concentration of the total base ions (OH_t%): OH_t%=0.99%.

6.1.3. Back Calculation of the Corporate Technique Standard from the Production Test Results

Back calculation coefficient=the measurement value of the primary material/corporate standard value=17.5%/9.2%=1.90

Corporate standard=measurement value/back calculation coefficient.

The corporate standard value of the primary material Fe_2O_3 =17.5%/1.90=9.21%.

The corporate standard value of the alkalizing agent MgO=1.4%/1.90=0.73%

The total base ion corporate standard value=0.99%/1.90=0.521%

6.1.4. Calculation of Moles of Ions

The corporate standard value can be converted to the related moles of ions (i=y, k, $[OH_t]$) by multiplying the mole conversion constant (ω_M) to the weight percent concentrations of each ion:

$$y=3.76(\omega_{Fe})\times 9.21\%=0.346$$

$$k=4.96(\omega_{Mg})\times 0.73\%=0.036$$

$$[OH_t]=5.89(\omega_{OH})\times 0.521\%=0.031$$

6.2. Calculation of the Alkalinity, Coagulation Strength, Acid Radical, and Bonding Water

6.2.1. Calculate Alkalinity (B%)

The alkalinity of basic metal ion:

$$B\%=[OH_t]/\sum i \quad \text{Where: } \sum i=y+k=0.382 \qquad (2)$$

Input the number of moles of related ions (i=y, k, $[OH_t]$) into the equation (2), we can obtain B%:

$$B\%=0.031/0.382=0.081=8\%$$

6.2.2. Calculate Coagulation Strength (C%)

$$C\%=[\sum M_i^{n\geq 3+}]/[\sum M_i^{n\geq 2+}]=[0.346/0.382]=90.6\%\geq 90\%$$

6.2.3. Calculate the Number of Moles of the Sulfate Radical [$(SO_4)_{(ni-niB)/2}$] That Combines the Basic Metal Ions

Input the number of moles of related ions and B value (i=y, k, B into the equation (1) and calculate

$$(SO_4)_{(3y-3yB)/2} \text{ and } (SO_4)_{(2k-2kB)/2}:$$

$$(SO_4)_{(3y-3yB)/2}=(SO_4)_{(1.038-0.083)/2=0.478}$$

$$(SO_4)_{(2k-2kB)/2}=(SO_4)_{(0.072-0.006)/2=0.033}$$

6.2.4. Calculate the Number of Moles (w) of the Bonding Water (wH_2O) in Hydrated Salt

Input the number of moles of related ions and B value (i=y, k, B value) in to the following equation and calculate the w:

$$w=6(y+k)-B(3y+2k)$$

$$w=6\times 0.382-8\%\times(3\times 0.346+2\times 0.036)$$

$$=2.30-0.09=2.21$$

6.3. The Molecular Formula of the Product

Use the above calculation results and input the values in to the equation (1) to obtain the product molecular formula ($\prod M_i$) as the expression listed below:

$$\prod M_i=[Fe_{0.346}(OH)_{0.083}(SO_4)_{0.478}Mg_{0.036}(OH)_{0.006}(SO_4)_{0.033}\cdot 2.21H_2O]_{m=1.90}$$

6.4. The Characteristics of the Product Liquid

(1) Appearance: dark brown; density (room temperature): 1.5(g/cm^3).

(2) Chemical ingredient: Fe_2O_3/17.5%; MgO/1.4%; OH_t/0.99%; silicone calcium residue/0.5%.

(3) Coagulation function: alkalinity B≈8%, coagulation strength C≥90%-grade II/medium strong.

(4) Molecular formula simplification [22]: the simplified molecular formula for poly-ferric magnesium sulfate is

$$[Fe_{0.346}(OH)_{0.083}(SO_4)_{0.478}\ldots\bullet 2.21H_2O]_{m=190}$$

(5) The name of the product [22]: using two monomers of the poly-basic ferric magnesium sulfate, it can be simply called “poly-ferric sulfate” or “PF…S”. Since it used as a coagulating agent, it can also be called “poly-

ferric sulfate coagulating agent" or "PF…S coagulating agent".

7. Conclusions

This paper suggests using ferrous sulfate as a primary material (oxidized to ferric sulfate by sodium chlorate) and magnesite powder as an alkalizing agent in a heating reaction pool to produce the coagulant PF(Mg)···S. This process consumes the lowest energy, obtains coagulant's alkalinity B≈8% and reaches high coagulating strength C≥90%- grade Ⅱ/ medium strong.

Competing Interests

The authors declare no conflict of interest.

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[i] the four-neck flask: 1 neck for an electric mixer, 1 neck for a straight condenser tube, 1 neck for thermometer, 1 neck for material addition or sample withdrawn for checking reaction completion status.

[ii] Fe^{2+} analysis method: withdraw a small amount of sample liquid, put 1 drop sample liquid in a test tube, add 1 drop of 10% potassium ferricyanide solution [$K_3Fe(CN)_6$]. If no blue precipitate showing up, that means no ferrous ion left in the reaction solution and the reaction is completed.