Optimization of Neutralize Crude Gypsum (NCG) Quality through Flash Dryer Utilization at Plant IIIA PT. Petrokimia Gresik

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Abstract

Neutralized Crude Gypsum (NCG) is produced as by-product from phosphoric acid plant at Plant IIIA PT. Petrokimia Gresik. The aims of this study are finding main cause and solution from Neutralized Crude Gypsum (NCG) quality reduction at PT. Petrokimia Gresik Plant IIIA. This research is done by qualitative analysis in order to collect the data of gypsum quality every month. Gypsum characteristics is observed by some instrument analysis such as spectrophotometry UV-Vis, atomic absorption spectrometry (AAS), and gravimetry technique. NCG was taken at different point on gypsum disposal land and different times to analyze its quality change. NCG sample shows decreased quality having higher moisture content at September that does not comply the SNI 715:2016 about gypsum minimum quality. Flash dryer is designed in this study to reduce the water concentration inside the surface of gypsum particle. Flash dryer design can lower the H₂O content based on material balance analysis at finishing sector Plant IIIA unit.

Keywords: flash dryer, gypsum, moisture, NCG

1. Introduction

PT. Petrokimia Gresik is one of the largest fertilizer production companies in Indonesia [1]. PT. Petrokimia Gresik has several plants producing a lot of fertilizer product [2]. Plant IIIA at PT. Petrokimia Gresik produces some products such as sulfuric acid, phosphate acid, ammonium sulphate usually known as ZA fertilizer, and gypsum [3]. Phosphate acid as by product can be processed at Plant IIIA until 610 ton/day with concentration of P₂O₅ approximately 54 %wt [4]. This phosphate acid is used as a raw material for phosphate-based fertilizer synthesis at Plant II [2].

Another by product from Plant IIIA is gypsum that also known as NCG (Neutralized Crude Gypsum) due to its neutral pH [5]. Gypsum commonly found as calcium sulfate dihydrate with chemical formula CaSO₄.2H₂O [6]. Gypsum usually used as mineral adhesive because of its superiority characteristics than another ordinary organic adhesive [7]. Gypsum was claimed more environmentally friendly, cheap, fireproof, and resistant to biological deterioration [8].

Gypsum is a kind of mineral with high calcium content used as an adhesive because of its hydrous characteristics for soil reinforcement [9]. Gypsum has chemical formula CaSO₄.2H₂O with molecular weight 172.17 gr/gmol. The chemical composition of gypsum is calcium (23,28%wt), calcium oxide (32,57%wt), water (20,93%wt), and sulfur (18,62%wt) [10]. Gypsum has a solubility in water creating suspension with specific settling time [11]. It is the times gypsum needed for being hard and compact measured from contact with water. Physically, gypsum has specific gravity
ranging from 2.31–2.35 and solubility in water 1.8 gr gypsum/liter at 0 °C of temperature [11].

NCG can be sold in bulk using 50 kg per sack, very interesting product for consumer as light brick raw material. NCG usually consists of an amount gypsum fulfilling light brick production standard listed in SNI 715:2016 about gypsum minimum quality for light brick [12]. With a high moisture content and a more agglomerate appearance, the quality of NCG deteriorated at September. NCG’s purity fell below 80%wt because its water content exceeded 20%wt, and it does not fulfill the gypsum criterion [13]. This circumstance is possible because Indonesia's rainy season often begins in mid – August and increases the air humidity of surrounding area [14]. This situation causes a 20% decline in NCG sales since it does not comply with SNI 715:2016 [1]. This study aims to identify the primary source of NCG quality decrease. The second purpose is to discover a means to optimize the process system or equipment at Plant IIIA PT. Petrokimia Gresik so that the NCG quality meets the SNI 715:2016 specifications every month.

2. Material and Method

This study was conducted by quantitative analysis of NCG (Neutral Crude Gypsum) properties at Plant IIIA PT. Petrokimia Gresik. The sample of NCG was collected and characterized from July to September. NCG sample was taken from three points at a depth of 1 meter in cement retender storage building Plant IIIA PT. Petrokimia Gresik (Figure 1). There are some laboratory analyses had been done to monitor the NCG quality such as moisture content analysis, sulfur concentration, and CaSO₄.2H₂O composition itself.

Water content inside gypsum was measured by gravimetry technique using drying process. Moisture content measurement had been conducted using two steps gravimetry method. The NCG sample was weighed before being heated at 100°C for four hours [15]. After that, the sample was weighed again so that the loss and the difference in weight obtained became Free H₂O (F.H₂O). The second step of this gravimetry analysis aims to check the real contained water inside gypsum (C.H₂O). The NCG sample then is heated until 250°C for 30 minutes and weighed again to measure the real H₂O molecules bonded with CaSO₄ [15]. Moreover, sulfur concentration as SO₃ groups was analyzed by Spectrophotometry UV-Vis apparatus. CaSO₄.2H₂O was characterized using Atomic Absorption Spectrometry (AAS) for molecular quantity interpretation. All of sample analysis had been finished at Chemical Laboratory of PT. Petrokimia Gresik.

Fig. 1. Sampling point at NCG Stock Building
3. Results and Discussion

NCG (Neutralized Crude Gypsum) is by-product of phosphoric acid process at Plant IIIA. Phosphoric acid plant has several unit processes like grinding unit, hemihydrate reaction unit, fluorine recovery unit, hemihydrate filtration, hydration unit, and dihydrate filtration [2]. Gypsum was firstly formed at hydration process and dihydrate filtration unit. Hydration process of hemihydrate cake was occurred as further reaction process of phosphoric rock residue and sulfuric acid via hydration tank. Dihydrate slurry, then, was separated at dihydrate filter yielding phosphor gypsum cake as by-product of phosphoric acid plant [1].

\[
\text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}
\]

\[
\text{Ca}_2(\text{PO}_4)_2 + \text{H}_2\text{SO}_4 \rightarrow \text{H}_2\text{PO}_4 + \text{CaSO}_4 \cdot 2\text{H}_2\text{O}
\]

NCG (Neutralized Crude Gypsum) is gypsum slag result drying naturally over two weeks on disposal ground. The desired moisture content in NCG after this final process is approximately 19%wt [5]. Afterwards, NCG was distributed into cement retender storage and would be added by captan (C_{6}H_{4}Cl\{NO_{3}\}) 3%wt in order to make its acidity near to neutral (minimum pH 6). NCG was analyzed periodically to find out its quality every time.

There are many factors occurred towards NCG product quality deficiency. Several internal problems at Plant IIIA were happened that is gypsum stack crisis on disposal due to plant shutdown over two months and utilization of NCG continuously for ZA-II production. NCG color changes darker and more agglomerate than usual due to its storage time more than six months on disposal land (Figure 1). Increasing light brick quality can be done by varying gypsum approximately 0-10 %wt because of its moisture content stability. It results that gypsum addition can enhance the water absorption inside light brick due to enlargement of bricks pore and unbalanced solution was occurred [5][16].

Water absorption test is conducted regarding SNI 15-2094-2000 that explaining maximum value of water absorption is 20 %wt [16]. Otherwise, gypsum addition ranging from 2,5-3 %wt can rise its compressive strength while more than 3 %wt can reduce its robustness because of its structure becomes more void increasing water absorption [7]. It can be happened because gypsum is one of easily gained and very effective stabilization material. Gypsum is one of mineral compound with high calcium concentration as soil and clay binding. Gypsum is also more hydrophobic that useful for strengthening the soft ground [12].

NCG was usually found more agglomerate, darker, and more watery than standard quality every after July (during rainy season). NCG analysis results (Figure 2) was obtained by collecting data at July and September. NCG test result approximately 41,4%wt of SO_{3} and 88,9%wt of CaSO_{4}.2H_{2}O concentration at September. On the other hand, NCG analysis result 43,4%wt of SO_{3} and 93,2%wt of CaSO_{4}.2H_{2}O concentration on July. Moisture content test result during July and September indicate stable value at 18,6%wt (July) and 19,5%wt (September).

NCG product is fulfilling the SNI 715:2016 at July shown by its moisture content below 19%wt as per SNI standard. Otherwise, the water

![Fig. 2. NCG analysis result](image)

![Fig. 3. Sampling test at three points (A, B, C)](image)
content of NCG has increased 0.5%wt during September. That enhancement makes the purity of CaSO$_4\cdot$2H$_2$O inside of NCG is falling down reach 88.9%wt, approximately 3%wt less than the SNI standard value. But there is no significantly change of SO$_3$ concentration of NCG during July and September satisfying the maximum standard of SNI.

Furthermore, NCG was also measured its water content at three points of sampling location in cement retender stockpile (Figure 1). Moisture content measurement had been conducted using two steps gravimetry method. NCG test result at three points of sampling shows various water content value. NCG product has 14.57%wt of free H$_2$O (F.H$_2$O) and 18.9%wt of C.H$_2$O complying the SNI Standard. Moreover, NCG sample at point B has 13%wt of F.H$_2$O and 19.18%wt of C.H$_2$O that does not meet the SNI standard. Besides that, NCG test result from sampling point C shows more satisfactory value that of 11.7%wt F.H$_2$O and 15.66%wt C.H$_2$O. NCG purity from point C is 74.83%wt drastically lower than sample from point A and B. Otherwise, NCG purity at point A and B are slightly different of 90.3%wt and 91.66%wt respectively. It is shown from Figure 3 that C.H$_2$O concentration is larger than F.H$_2$O content at all sampling A, B, C point.

C.H$_2$O amount is obviously more than F.H$_2$O concentration in the bulk of gypsum at all of sampling location (A, B, C). Therefore, NCG (Neutralized Crude Gypsum) natural drying process on ordinary disposal land is not enough to reduce the C.H$_2$O content. So that, the NCG products at September which is starting to rainy season have higher amount of H$_2$O molecule than usual. It needs more drying step process using drying equipment to decrease its water content before mixing with captan compound 3%wt [16].

Flash dryer is proposed to maximize drying process based on reference from APV Dryer Handbook. Flash dryer could produce high drying
rate approximately 250 until 1250 kg/m³/h [17]. Moreover, this equipment needs a little space than other drying apparatus saving more area in the production yard [18]. Flash dryer has been equipped by blower to transport hot air from heater to drying vessel or usually called as flash duct (Figure 4). Hot air acts as heating media to speed up water molecules evaporation from inside bulk of gypsum into atmosphere [19]. Afterwards, the dried gypsum is pushed into cyclone as separation apparatus for particle – air separation. A little amount of fined particle is carried away with hot air and trapped inside the bag filter. So, the discharged hot air is free of particle minimizing the air pollution and moves to stack [20].

Based on collecting data, PT. Petrokimia Gresik Plant IIIA could produce gypsum as NCG approximately 140,000 ton/year. So that, flash dryer is calculated having 16 ton/hour capacity. Flash dryer with this capacity can reduce water concentration inside gypsum from 19,18 %wt into 15 %wt [19]. Furthermore, water molecules that evaporate from gypsum surface to atmosphere approximately 1128,024 kg/hour with drying efficiency 93%.

4. Conclusions

Neutralized Crude Gypsum (NCG) quality from Plant IIIA PT. Petrokimia Gresik at September was decreased significantly with darker and more agglomerate apparition. This can be happened due to gypsum stock crisis at disposal land as a result of plant shutdown. Therefore, NCG product looks like a boulder because contains higher moisture content especially H2O molecules that bonded into CaSO4 particle as crystalline structure. Flash dryer is proposed to reduce the water concentration before gypsum is mixed with captan as an additive. Flash dryer is successfully lower the moisture content regarding mass balance calculation at gypsum processing system.

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