
Effects of Extraction Temperature and Time on the Physical Properties of Soluble Sodium Silicate from Rice Husk Ash

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Abstract: In this research, soluble sodium silicate was extracted from rice husk ash. Rice husk in its as-received state was initially burnt in an open air to obtain the rice husk ash (RHA), which was later thermally treated inside the muffle furnace at 600°C for 5hrs to remove the carbonaceous matter and increase the silica (SiO₂) content. The thermally treated RHA was later mixed with 3M NaOH as soda source and heated with continuous stirring inside a heating glass vessel placed in a thermostatic water bath at 70°C for 4hrs, 80°C for 3hrs, and 90°C for 2hrs respectively. Physical analyses such as Viscosity, PH, Specific gravity, and Electrical conductivity were carried out on the extracted sodium silicate which was compared with the reference sodium silicate sample.

Keywords: Rice Husk Ash (RHA), Sodium Silicate, Viscosity, PH, Specific Gravity

1. Introduction

Sodium silicate solutions are complex mixtures of silicate anions and polymer silicate particles of SiO₂: Na₂O molar ratio >2 which are commercially referred to as water glass [1]. The process of producing Na₂SiO₃ comprises basically four main stages as stated by [2]: (1) calcinations of a mixture of Na₂CO₃ and SiO₂ in furnace at 1400°C -1500°C to obtain a solid glass; (2) Dissolution of the solid glass produced in a reaction vessel subjected to high pressure and temperature to produced Na₂SiO₃ solution and impurities (not reacted silica); (3) Optimal filtration depending on the purity desired, (4) Evaporation of water from the silicate solution for the production of solid sodium silicate. This process is however considered to be very expensive due to the high energy required to achieve a high temperature calcination reactions in addition to generation of air pollution from emission of dust particles, SO₂, and Nitrogen.

The calcination process is mostly used in industrial production of sodium silicate; there is therefore another process with least energy required and no issue with dust emission which involves reaction of silica with aqueous NaOH in an autoclave under temperature and pressure or in

an open system under atmospheric pressure as stated by several researchers and patents [2-7, 9]. [8] However reported that silica (SiO₂) is available in large amount in soils and are accumulated in the tissues of root plants, and therefore, several agricultural wastes such as corn cobs, wheat husks, rice husks, bagasses etc. are potential source of silica which on ash can be used as silica source. Critical economic and environmental situations in recent times have therefore led industries and researchers to develop and improve technologies meant to drastically minimize industrial wastes. In view of this, many efforts have been concentrated in several areas, including agricultural production [9].

Rice is the second most produced cereal in the world with the estimation of approximately 40 million tons of milled rice in 2003 [10]. Production of rice is geographically concentrated with Asia having over 90% of the world output while country like Nigeria is putting efforts to develop this sector of the agriculture produce.

In Nigeria, most rice husks generated are mostly sent to landfill or burnt as fuel to generate energy which therefore leads to the production of residual ashes (RHA) containing over 60% silica content. Rice husk ash (RHA) has therefore been considered as a new economically potential source of silica [11].

There have been numerous researches on the utilization of rice husk for manufacture of silica based compounds such as silicon carbide, silica gel, silicon nitride, xerogels, and pure silica [12-17]. [13] Discovered a new, lower energy required process of producing sodium silicate from rice husk ash. Their study showed that silica available in the rice husk ash was amorphous at 600°C which was reacted with NaOH to obtain sodium silicate solution. [17] employed the method utilized by [12] to synthesis sodium silicate from rice husk ash containing 77% SiO₂; which was later reacted with magnesium sulfate salt at room temperature to obtain magnesium silicate. [3] Have also studied the conversion of rice hull ash into soluble sodium silicate. In their study, RHA was reacted with NaOH solution under open and closed systems. In open system, reaction was carried out in a glass reaction vessel under atmospheric pressure while the closed system was carried out under temperature and pressure inside an autoclave.

This present work aimed to produce soluble sodium silicate from rice husk ash at different temperature and time interval employing the adapted method stated by [16]. The effects of these parameters (temperature and time variations) on the properties such as viscosity, PH, specific gravity, electrical conductivity, and the characteristics of the sodium silicate produced were investigated.

2. Materials and Method

2.1. Combustion and Heat Treatment of Rice Husk Ash (RHA)

The collected rice husks were first washed to remove dirt and other sand particles associated with the husks; and dried. After drying it was placed inside a perforated cylinder pan for combustion into ash. 120gram of the obtained ash was then placed inside an alumina crucible placed inside a muffle furnace and heated at a temperature of 600°C for 5hrs to reduce the carbonaceous matter and increase the percentage of active silica content and obtained amorphous silica phase as stated by [13].

2.2. Chemical Analysis of the Rice Husk Ash (RHA)

The chemical analysis was carried out using thermo-gravimetric analysis. This was carried out as stated in the Annual Book of the ASTM standard (ASTM, 1986).

0.5g of the RHA was mixed thoroughly with 0.5g of NH₄Cl in a 50ml beaker. The beaker was covered with a watch glass and a 5ml of HCl was cautiously added by allowing the acid to run down the lip of the covered beaker. Then 1-2 drops of HNO₃ was added to the mixture after chemical reaction has subsided and stirred with a glass rod. The covered beaker was set on a steam bath for digestion for 30mins with occasionally stirring and breaking up the lumps to facilitate complete decomposition of the feldspar. A Whatman No. 41 ashless filter paper was used to filtrate a jelly-like mass of silicic acid as completely as possible without dilution. The beaker was rinsed with hot HCl, 2-3

times and then with 10-12 small portions of hot water. Each portion is allowed to drain through completely before adding another. The filtrate was reserve for determination of ammonium hydroxide group. The filter paper and the residue were transferred into weighed platinum crucible, W1. This material was dried and ignited slowly without flaming until filter paper was charred and finally at 1100-1200°C for 1hr in a muffle furnace. This material was cooled and weighed, W2. The SiO₂ obtained contains small impurities which were treated in the crucible with 1 or 2ml of H₂O, 2 drops of H₂SO₄ and 20ml of HF and cautiously evaporated to dryness. The residue was heated at 1050°C for 5mins, and cooled and weighed W3.

$$\text{SiO}_2 (\%) = [(W1 - W2) + W3] / W \times 100$$

W = weight of sample taken

W1 = weight of silica and insoluble impurities

W2 = weight of impurities

W3 = weight of silica recovered from iron and aluminum oxide

2.3. Weighing of the Rice Husk Ash

Three samples A, B, and C measuring 30 gram each was weighed from the obtained heat treated rice husk ash which were later reacted with concentrated sodium hydroxide and boiled in an heating glass vessels at temperature of 70°C, 80°C, 90°C for 2hrs, 3hrs, and 4hrs respectively.

2.4. Preparation of Sodium Hydroxide Solution

3M NaOH was prepared to react with the RHA which was carried out by calculating the molarity of the NaOH pellet using the formula below.

$$\text{Molarity} = \text{mass} / \text{molar mass} \times 1 / \text{volume}$$

$$\text{Molar mass of NaOH} = 32 + 16 + 1 = 40.$$

$$3 = \text{mass} / 40 \times 1 / 1$$

$$\text{Mass} = 3 \times 40 = 120\text{g}.$$

This means 120g of NaOH Pellets will be dissolved in one liter of distilled water. This was obtained by dissolving the sodium hydroxide pellets inside a 500ml conical flask containing distilled water using stirring rod to stir it until the pellets were totally dissolved.

2.5. Reaction in the Heating Bath

The reaction was carried out in accordance with the procedure stated by [12]. 180ml portion of the 3M NaOH was poured inside 500ml Erlenmeyer flask containing 30 gram weighed sample of RHA of samples A, B, and C placed respectively inside a thermostatic heating bath and the temperatures were set to 70°C, 80°C, 90°C for 2hrs, 3hrs, and 4hrs respectively with continuous stirring. The solutions were afterward allowed to cool down to room temperature after the boiling process. The solutions obtained from samples A, B, and C were then filtered separately using a Whatman No. 41 filter paper into separate 250ml round bottom flask while the residue remaining on the filter paper were discarded.

3. Results and Discussion

3.1. Characterization of the Rice Husk Ash

The results of the analysis carried out on the rice husk ash (RHA) shows that the inorganic content of this ash contains a good percentage amount of SiO_2 (82%) and Alumina content of 4%. The high silica content therefore justifies the use of the rice husk ash (RHA) as a silica source for this research work.

3.2. Analyses of the Produced Na_2SiO_3

Table 1 shows the results for the various analyses carried out on the produced sodium silicate samples A, B, and C which were compared with the standard sample as a basis of reference. The results revealed that PH values for samples A, B, and C ranges from 12.8-12.9, 12.7- 12.8, and 12.7- 12.9 respectively while the reference sample has a PH value ranges from 8-13.0. The specific gravities are 0.8035, 0.7944, and 0.7803 respectively while the electrical conductivities are

911 $\mu\text{s}/\text{cm}$ and 744 $\mu\text{s}/\text{cm}$, and 745 $\mu\text{s}/\text{cm}$ for the three samples respectively. The samples B and C have viscosities of 400 centipoises while sample A has a viscosity of 280 centipoises and the reference samples has the highest viscosity of 830 centipoises. All the produced samples exhibited characteristic syrupy, high alkalinity liquid which conforms to the reference sample.

3.3. Clay Deflocculation Test

Few drops of the produced sodium silicate samples A, B, C, and the reference sample were added to already prepared clay slip to determine From the clay deflocculation test, it was observed that sample A required more drops before the effect can be seen in the clay slip compare to sample B. Both samples however works in deflocculation but not as potent as the reference sample. This might however be attributed to the low temperature employed in this research compare to reference sodium silicate produced industrially.

Table 1. Analyses of the Produced Sodium Silicate and Reference Sodium Silicate.

Samples	PH Value	Electrical conductivity ($\mu\text{s}/\text{cm}$)	Specific Gravity (g/cm^3)	Viscosity (centipoises)	Characteristics
Sample A at 70°C for 4 hrs	12.8-12.9	911	0.8035	280	Syrupy liquid, high alkalinity
Sample B at 80°C for 3 hrs	12.7 -12.8	744	0.7944	400	Syrupy, alkaline liquid
Sample C at 90°C for 2 hrs	12.7 -12.9	745	0.7803	400	Syrupy, alkaline liquid
Reference Sample	8 – 13.0	-	1.39	830	Syrupy liquid

4. Conclusion

The following conclusion can be drawn within the limit of this work:

- Sodium silicate can be extracted from rice husk ash as a silica source
- The extracted soluble sodium silicate displayed lesser viscosity as compared with standard sample which may be attributed to the low temperature of extraction
- The extracted soluble sodium silicate has displayed similar physical characteristics as compared to the standard sample.

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