OPTIMIZATION OF THE PRODUCTION OF SODIUM SILICATE FROM RICE HUSK ASH

ADAMU, M.[1]; OKAFOR, J .O.[2] & DAVID, G. T.[3]
[1,3]Department of Chemical Engineering,
Federal Polytechnic, Bida, Niger State
[2]Department of Chemical Engineering,
Federal University of Technology, Minna, Niger State
E-mail: mudams2004@gmail.com Phone No: +234-806-529-5882

Abstract
The conversion of rice husk which to sodium silicate was investigated. The rice husk was dried and ashed at various temperatures of 550°C, 600°C, 650°C, 700°C and 750°C. 25g of 0.15 mm size range of each sample was reacted with 0.5M sodium hydroxide solution at 80°C for 60 minutes. The average yields of sodium silicate obtained were: 2.20g, 3.25g, 5.45g, 5.15g and 5.02g respectively. Using the sample of ash produced at 650°C, the production process was optimized.The reaction temperature was varied using 80, 90, 100, 110, 120, 130 and 140 °C and optimum yield of 5.25 g sodium silicate was obtained at 120 ° C at a fixed concentration of sodium hydroxide solution and reaction time of 0.5 M and 30 minutes respectively. When the concentration of sodium hydroxide and reaction time were varied, the optimum yield of 19.80 g and 39.4 g sodium silicate were obtained at 2.5 M and 60 minutes respectively. From the results obtained, the optimum yield of 39.4 g sodium silicate was achieved at optimum operating conditions of temperature, time and concentration of 120 °C, 60 minutes and 2.5 M respectively. Some confirmatory tests were carried out to ascertain the presence and quality of the sodium silicate produced. The % Na_2O, %SiO_2, %H_2O and weight ratio of SiO_2/Na_2O were found to be 8.2%, 27.3%, 64.5% and 3.3 respectively while the specific gravity, viscosity and pH are 1.392, 198.52 and 12.2 respectively.These values are in close agreement with those obtained from a commercial grade sodium silicate. It can be inferred from the results of this study that production of sodium silicate from rice husk is possible.

Keywords: Optimization, Sodium, Silicate, Rice Husk, Ash

Introduction
Critical economic and environmental situations ofthe current days encouraged companies and researchers todevelop and improve technologies intended to reduce orminimize industrial wastes. As a consequence, much efforthas been expended in different areas, including the agricultural production (Ana Maria *et al*., 2009).

Rice is the second largest produced cereal in theworld. Its production is geographically concentrated in Asia withmore than 90 percent ofworld output (Real *et al*., 1996). The United States and Brazil are the most important non-Asianproducers and Italy ranks first in Europe. The rice worldproduction was approximately 400 million tons of milled rice in 2003 (Craig *et al*., 2010).

Rice husks makes up about 20% of the rice (paddy) weight. The rice millersworldwide thereforeshould generate more than 100 million tons of rice husks. Nowadaysalmost 70% of the rice husks are not commercially used (David, 2011).

All over the world the discharge of the husk accruing in the numerous ricemills causes a serious environmental problem. This agricultural waste howeverhas a significant calorific value and a high percentage of amorphoussilica. With an innovative technology these favorable characteristics of rice huskwill be used for solving the environmental problems and at the sametime producing electricity and high value industrial products (David, 2011).

Burning rice husk as fuel to generate energyresulted in a waste product, namely rice husk ash. Rice husk ash is rich in silica (92-97%) and can bean economically viable raw material for production of silica gel and silica compounds (Supitcha *et al.*, 2009).

Soluble sodium silicates (or water glass) are solutions of water and soluble glasses manufactured from varied proportions of $Na_2CO_3$and SiO_2 (Craig & Heather, 2010). Liquid Sodium Silicate is used to manufacture laundry soap, detergent cake, Sodium meta-silicate, fire resisting and acid proof cement, steel and alloy castings, electrodes, dyeing, printing, textile auxiliaries and ceramics. Because of its unique properties, sodium silicate is used in various applications to manufacture zeolite, construction chemicals, oil field chemicals, paints, pigments, paper making, paper cones, tubes, foundry, fluxes and fiber drum. Because of its binding properties Sodium Silicate is the best water proofing agent and it is also used in acid and alkali proof linings (Craig & Heather, 2010).

Optimization of the reaction variable allows obtaining the best conditions necessary to produce sodium silicate. The traditional way of achieving this task is the ‘one variable at a time’ approach. This traditional method involves keeping other variables constant and varying the one under test in other to find it’s value at optimum yield (Yisa, 2011).

In order to utilize the potentials of rice husk ash produced from rice husk and to also solve environmental problems, this research is aimed at converting rice husk which is an agricultural waste to sodium silicate which is a raw material for many chemical processes.

Materials and Methods

Source of raw material and reagents: The raw material rice husk was obtained at a dumping site at Takowasa area in Bida, Niger state. The rice husk was washed to remove the dirt and sun dried to remove the moisture. The reagents used are NaOH pellets and Dilute Tetraoxosulphate (VI) acid.

Methodology

Ashing of rice husk: A known mass of 1000 g of dried rice husk was ashed in a muffle furnace in five batches of 200g per batch and the temperature set at 550^{o}C for 24 hours for each batch. After each batch the content of the furnace was then discharged. The ashing process was repeated using different ashing temperature of 600^{o}C, 650^{o}C, 700^{o}C and 750^{o}C (Novotny*et al.*, 1991).

Preparation of standard solutions of NaOH: Electronic weighing balance (Adventure ARRW 60) was used to weigh 20g of NaOH pellets which was then placed in a 1500 ml beaker. Distilled water was added to it up to 1000 ml mark to give 0.5M. The resulting solution was properly stirred and stored in a plastic container. The procedure was repeated by dissolving 40g, 60g, 80g, 100g, 120g and 140g in the same quantity of distilled water to obtain 1.0M, 1.5M, 2.0M, 2.5M, 3.0M and 3.5M NaOH solutions respectively.

Effects of ashing temperature of rice husk on the yield of sodium silicate: A known mass of 25g of rice husk ashed at 550°C was placed in a stainless steel pot and 600 ml of 0.5M NaOH solution was added to it. The pot and its content were then transferred to a hot plate operating at 80°C. The mixture was continuously stirred for 60 minutes. The mixture was then allowed to cool after which it was filtered using filtration apparatus. The filtrate obtained was aviscous, transparent colourless, solution. The solution was then concentrated in an oven (GallikampSG 97-03-243) at 100°C for one hour. From the concentrated solution, the mass of sodium silicate produced was determined and recorded. The experiment was repeated with samples of rice husk ashed at 600°C, 650°C, 700°C and 750°C. Using the sample of rice husk ashed at 650°C, the above procedure was repeated in all the experiments carried outin optimization of the reaction variables for the production of sodium silicate from rice husk ash (Kalapathy*et al*., 2000).

Optimization of the yield of sodium silicate from rice husk ash: The major reaction parameters that affect the yield of sodium silicate from rice husk ash are: concentration, temperature, and reaction time. It is therefore paramount to alter these process variables for the purpose of optimizing the yield.

Effect of reaction temperature on the yield of sodium silicate: The effect of reaction temperature on the yield of sodium silicate was investigated at ifferent temperature of 80, 90, 100, 110, 120, 130 and 140 °C, while the concentration of Sodium hydroxide and reaction time were fixed at 0.5M and 30 minutes respectively. At the end of eachexperiment, the yield of sodium silicate was measured and recorded (Akpan *et al*., 2005).

Effect of concentration of sodium hydroxide on the yield of sodium silicate: The concentration of sodium hydroxide was varied using 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 and 3.5 M, while the temperature of reaction and reaction time were fixed at 120 °C and 30 minutes respectively. At the end of each investigation, the yield of sodium silicate was determined and recorded (Akpan *et al.,* 2005).

Effect of reaction time on the yield of sodium silicate: The reaction time of 30, 45, 60, 75, 90 and 105 minutes were used for the investigation, while the temperature of reaction and concentration of sodium hydroxide were fixed at 120 °C and 2.5 M respectively. At the end of each investigation, the yield of sodium silicate was determined and recorded (Akpan *et al.* 2005).

Confirmatory test for sodium silicate solution: Some standard tests were carried out on the final product produced to confirm the presence and the quality of sodium silicate after which the results were compared with those of a commercial grade sodium silicate.

Determination of percentage sodium oxide (%Na_2O): A known mass of 25 g of Commercial grade sodium silicate solution was diluted to 500 ml with de-ionized water. A 50 ml Aliquot was then titrated with 0.2 M Hcl until the pH of the mixture Become 4.3. The average titre value was calculated and recorded. This procedure was repeated using the sample of sodium silicate produced. In each case, the % Na_2O was determined using equation 1(Oxychem, 2002).

$$\% N_2O = \frac{\text{Titre value of Hcl} \times \text{Normality of Hcl} \times 3.1}{\text{Weight of silicate sample}} \quad (1)$$

Determination of percentage silica (% SiO_2): About 10 g of the commercial grade sodium silicate sample was diluted to 50 ml with distilled water. The resulting solution was then acidified with 200 ml of 0.2 M Hcl acid. The mixture was then evaporated todryness on a hot plate. The solid obtained was rinsed with distilled water to remove possible chlorides after which it was then ignited in a muffle furnace for 15 minutes. The final residue was then weighed and the % SiO_2 was calculated using equation 2 (Oxychem, 2002).

$$\% SiO_2 = \frac{\text{Mass of final Residue}}{\text{Weight of initial silicate sample}} \times 100 \quad (2)$$

Determination of percentage water (% H_2O): The percentage water content was calculated by the difference method using equation 3 (Gregory, 2005).

$$\% H_2O = 100 - (\% Na_2 + \% SiO_2) \quad (3)$$

Determination of pH, Viscosity and Specific gravity: The pH, Viscosity and Specific gravity of both the commercial grade and the sodium silicate produced were measured using pH mater, Viscometer and Hydrometer respectively (Oxychem, 2002).

Results and Discussion

The effect of ashing temperature on the yield of sodium silicate from rice husk was carried out due to the fact that the presence of crystalline or amorphous silica or both in rice husk ash depends on the burning temperature at which the ash is produced as reported by Yalcin and sevinc (2001). Considering the result obtained in Figure 1.0, the average mass of silicate obtained from rice husk ash produced at 550 ^oC was 2.2 g. As the temperature increases by 50 ^oC, the mass of silicate obtained was 3.25 g. The ashes produced at temperatures of 650 ^oC, 700 ^oC and 750 ^oC yielded 5.45, 5.15 and 5.02 g of sodium silicate respectively. As can be seen, the yield of Na_2SiO_3 increases sharply from ashes produced at temperature range of 550 to 650 ^oC after which the rate of increase in yield declined from the ashes produced at temperatures of 700 ^oC and 750 ^oC.

Since amorphous silica is more soluble than crystalline silica it can be deduced from this result that ashing temperature of 650 ^oC produced more amorphous silica which favour the yield of Na_2SiO_3. Ashing temperatures above 650 ^oC favour the production of crystalline silica which hinders the yield of Na_2SiO_3.

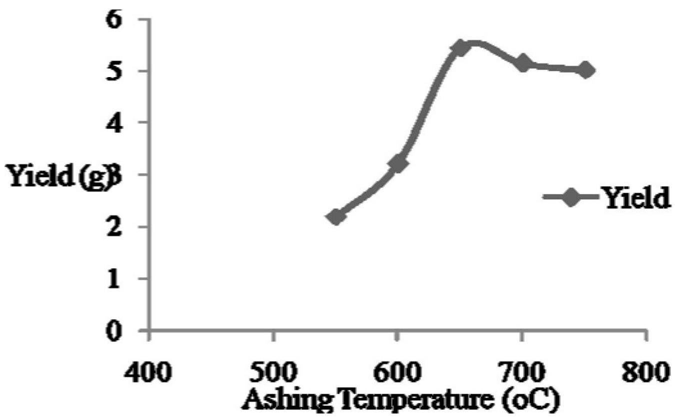

Figure 1: Effects of ashing temperature on the yield of sodium

The optimization of the yield of sodium silicate from rice husk ash was conducted using the rice husk ashed at 650^oC during which the three major reaction parameters which are temperature, concentration and reaction time were varied for the purpose of obtaining optimum conditions.The effect of reaction temperature was put into test by fixing the reaction time and the concentration of sodium hydroxide at 30 minutes and 0.5 M respectively while the reaction temperature was varied between 80 to 140 ^oC using a step change of 10 ^oC. The result obtained presented as shown in Figure 2. As can be deduced from the curve, the yields of sodium silicate increase significantly with the increase in reaction temperature range of 80 to 120 ^oC. At the reaction temperature of 80 ^oC the yield was 1.4 g, while the yields at 90, 100, 110 and 120 ^oC are 2.25, 3.35, 4.25, and 5.25 g respectively. The effect of temperature on the yield diminishes in vigor at temperatures of 130 and 140 ^oC with numerical values of 5.45 and 5.65 g respectively. From this result, the temperature of 120 ^oC can be selected as the optimum for the optimal yield of sodium silicate.

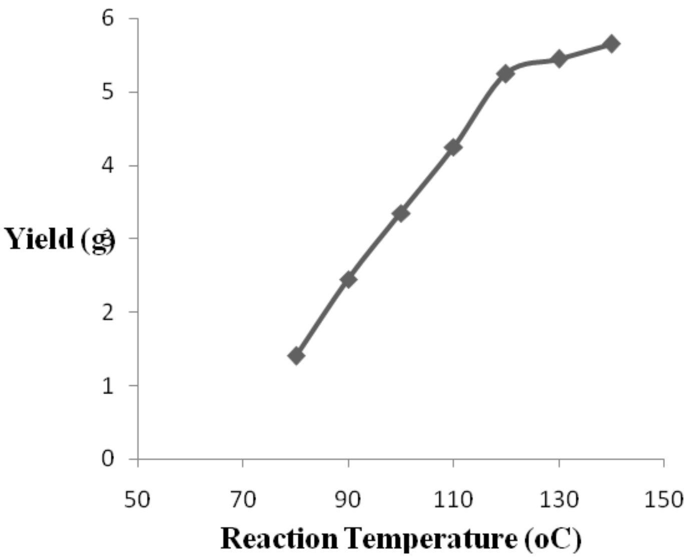

Figure 2: Effects of reaction temperature on the yield of sodium silicate

The effect of concentration of sodium hydroxide on the yield of sodium silicate from rice husk ash was investigated keeping the reaction temperature and time constant at 120 ^oC and 30 minutes respectively. As expected, the yield of sodium silicate increases with increase in concentration of sodium hydroxide. Referring to Figure 3, significant increase in yield was observed between the sodium hydroxide concentrations ranges of 0.5 to 2.5 M with values of the yields being 5.25, 8.80, 11.65, 16.15 and 19.80g at sodium hydroxide concentrations of 0.5, 1.0, 1.5, 2.0 and 2.5 M respectively. At sodium hydroxide concentrations above 2.5 M, the increase in yield was not significant. At NaOH concentrations of 3.0 and 3.5 M, the yields of silicate were 20.0 and 20.3 g respectively. At these concentrations, the amount of NaOH is in excess of what is required for the production of silicate from the limiting reactant which is the rice husk ash (RHA). By this result NaOH concentration of 2.5M is considered more favorable for the production process.

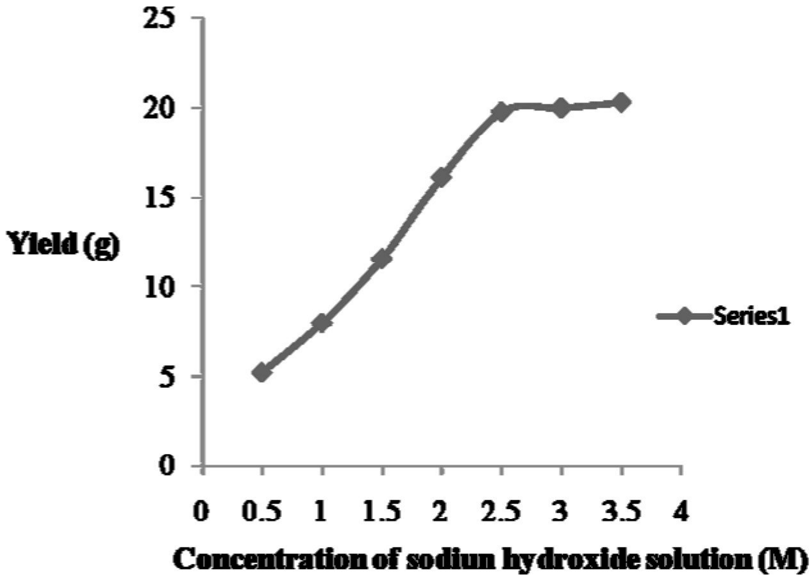

Figure 3: Effects of concentrationof sodium hydroxide on the yield of sodium silicate

The reaction time was varied with the aim of determining the suitable time that will give the optimal yield at the optimum conditions of temperature and concentration earlier investigated which are 120 ^{o}C and 2.5 M respectively. From Figure 4 it was observed from the curve that the yields increased vigorously between 30 to 60 minutes of reaction after which the rate of increase in yield declined between 75 to 105 minutes. This decline in the rate of increase in yield between 75 to 105 minutes may be attributed to the fact that the concentration of silica in the ash (The limiting reactant) could have drastically reduced due it being converted to sodium silicate before these reaction times. So therefore, the reaction time of 60 minutes can be considered more appropriate for the optimum yield at the optimum conditions of temperature and concentration.

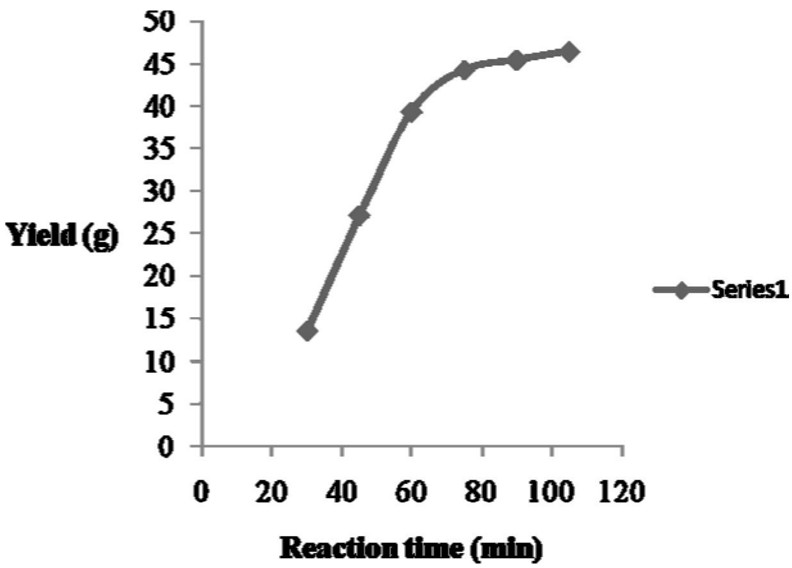

Figure 4: Effects of reaction time on the yield of sodium silicate

Some standard tests were carried out on the sodium silicate produced to confirm it presence and quality by comparing the results with that obtained from a commercial grade sodium silicate. The results of these tests are showed on Table 1. As can be seen from the results, the percentage Na_2O, SiO_2 and H_2O for the sodium silicate produced are: 8.2, 27.3 and 64.5 % respectively while that f the commercial grade was 9.1, 29.2 and 61.7 % respectively. When these results are compared it can be deducted that the sodium silicate produced is more diluted than the commercial grade due to higher percentage of water content. On the other hand, when the SiO_2/Na_2O ratio were compared, both samples falls within the standard range of 1.6-3.3 with the commercial grade having SiO_2/Na_2O ratio of 3 .22 and the sample produced from rice husk ash with a ratio of 3.3. When the specific gravities were compared, both have a value of approximately 1.4. The viscosity of 198.52 centipoises for the silicate produced is lower than the 206.25 recorded for the commercial grade. This may be attributed to the higer percentage of water in the sample produced. The pH recorded for the commercial grade was 11.3 while that of the sample produced was 12.2.

Conclusion

The effect of ashing temperature on the yield of Na_2SiO_3 shows that more amorphous silica which is more soluble than crystalline silica was produced at temperature of 650^oC thus giving the highest yield of sodium silicate. The production process was successfully optimized and the reaction variables optimized includes: reaction temperature, sodium hydroxide concentration and reaction time with optimum values of 120 ^oC, 2.5 M and 60 minutes respectively.

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