



Synthesis, Optimization and Characterization of Silica from Sugar Cane Bagasse and Rice Husk

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ABSTRACT

Silica was synthesized from Sugar cane bagasse and rice husk (Agricultural wastes). The pretreated samples were ashed and reacted with sodium hydroxide to produce sodium silicate from which silica was precipitated using Tetraoxosulphate (VI) acid. The optimum yield of silica from sugar cane bagasse was obtained at ashing temperature of 700 OC, ashing time of 2 hours, sodium hydroxide concentration of 4M, reaction temperature of 100 OC and reaction time of 2 hours. For the rice husk, the optimum parameters at which the optimum yield of silica was recorded are: Ashing temperature and ashing time of 700 OC and 3 hours respectively. For the reaction parameters which are: sodium hydroxide concentration, reaction temperature and reaction time, the optimum values were 3M, 100 OC and 3 hours respectively. The yield of silica from sugar cane bagasse at optimum conditions was 37.4% while that of rice husk was 66.3 %. The characterization of sugar cane bagasse ash, rice husk ash and the final silica obtained from both ashes all at optimum conditions were done using X-ray fluorescence (XRF). The result of the characterization indicated that sugarcane bagasse ash contains 64.01 % silica oxide (SiO₂) while rice husk ash contains 91.02 % silica oxide. The final silica precipitated from the sugar cane bagasse ash contains 99.03 % silica oxide while that from rice husk ash was 99.15 % oxide. These results indicated that significant quantity of amorphous silica can be successfully produced from rice husk and sugar cane bagasse..

Keywords: Agricultural wastes, bagasse, husk and silica

INTRODUCTION

The production of rice in the world in the year 2015 was estimated to be over 750 million tons (FAO, 2015). About 22% by mass of the rice produced is the husk which is about 154 million tons (Beagle, 1995). The husk contains 35 % Cellulose, 25 % Hemicellulose, 20 % Lignin, 3 % Crude protein and 17 % Ash. This statistic suggest that our planet is in an alarming rate being filled up with this Agricultural by-product and among all the cereal by-products, the rice hull contain the lowest percentage of digestible nutrients which amount to less than 10%. Among all cereal by-products it contains the lowest amount of protein and available carbohydrates (Olivier, 2004). Some of the applications of rice husk includes: pozzolan in cement industry (de-Gutierrez and Delvasto, 1994), aerogels (Nayak and Bera, 2009; Tang and Wang, 2005) activated carbon (Guo *et al.*, 2005), zeolites (Panpa and Jinawath, 2009) and cordierite (Chandrasekhar *et al.*, 2006).

When rice husk is incinerated its ash content is obtained as the product which contain about 90-98% amorphous silica. The silica content can be chemically retrieved by the action of suitable Alkali and finally precipitated using a mineral acid. Silica has variety of uses as catalyst support, filler, adsorbent and a raw material for synthesizing silicon and its compounds (Lorenzini, Biserni and Flacco, 2010).

Sugarcane cultivation is one the major agricultural undertakings in developing countries such as Nigeria. Every ton of sugarcane comprises of 142 kg cane juice, 140 kg of fiber (Bagasse), and the remaining being Agricultural residues with an average energy content of 15.7 MJ kg⁻¹ on dry basis (Alonso-Pippo *et al.*, 2008). The fibrous portion

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(bagasse) has chemical composition of 27–50 % cellulose, 20–35 % hemicellulose, 10–25 % lignin, and 1–6 % ash (on dry basis) (Chandel *et al.*, 2013).

In Nigeria, sugar cane bagasse is not fully utilized as a result; most of it is dumped as waste. Sugar producing countries like Brazil utilizes the advantage of bagasse combined heat and power generation estimated to be 8000 kJ kg⁻¹ dried bagasse used in boilers to generate electricity (Teixeira *et al.*, 2008). Combustion of about one ton of sugarcane bagasse produces about 60 kg of ash (Balakrishnan and Batra, 2011). Based on this statistics, it can be concluded that several thousands of tons of fly ash are wasted into the atmosphere (airborne fly ash) yearly which poses a threat to human health (Teixeira *et al.*, 2010). In most of the sugarcane industry, the boilers used operate on suspended grate combustion systems. In this system, the black fumes produced are evolved through the chimney while the ash is precipitated to the bottom (Brito *et al.*, 2003; Codeiros *et al.*, 2009). The fly and bottom ashes has composition (on dry basis) of 10–30 % char and 70–90 % inorganic matter mainly SiO₂ (silica) followed by CaO and others (Souza *et al.*, 2011). By this composition of the ash, silica can also be retrieved from it using a strong base and finally precipitated using an acid.

This research work is therefore aimed at optimizing the synthesis of silica oxide from rice husk and sugar cane bagasse and to characterize the ash and the final products produced at optimum parameters.

METHODOLOGY

The raw materials used for this study were Agricultural wastes; sugar cane bagasse and rice husk. The rice husk was obtained from a dumping site in Bida Niger state, Nigeria. While the bagasse was obtained from a fresh sugar cane bought from the market, Bida, Niger State, Nigeria.

Raw sugarcane was obtained from the market and the back removed using knife. The remaining part of the sugarcane was introduced into a thresher inside which the juice was squeezed out to obtain the bagasse. The bagasse so obtained was washed several times with distilled water until significant traces of sugar was removed. The bagasse was dried, sized reduced to 300µm. The sized sample obtained

was then treated with 3M HCl at 100°C for 2hours in order to reduce its aluminum and iron content. After the treatment, the treated bagasse was washed with de-ionized water until the pH of 7 was achieved. The samples were dried in an oven at 100°C until a constant weights were obtained. The same procedure was carried out using rice husk.

Variation of ashing temperature on the yield of silica oxide from samples

100g of dried treated bagasse ash was placed in a furnace set at 500°C for 2hours after which it was allowed to cool. The ash was then crushed and sieved to a workable size of 75µm. 10g of the ash was treated with 75ml of 1M NaOH solution at 100°C for 1 hour. At the end of the allotted time, the content of the beaker was filtered. The filtrate was titrated against 6.0M H₂SO₄ until a pH 7 was obtained. The resulting solution was then filtered and the white precipitate obtained (silica) was washed several times with distilled water after which it was dried in an oven at 80°C until a constant weight was obtained. The weight of the final sample was recorded as the yield of silica. The above procedure was repeated by varying the temperature of ashing using 550, 600, 650, 700, 750 and 800°C. The whole procedure was then repeated using rice husk (Adamu, *et al.*, 2014).

Variation of ashing time on the yield of silica oxide from samples

By fixing the ashing temperatures of sugar cane bagasse and rice husk that gave the optimum values, the ashing time were varied using 1,2,3,4 and 5hours and at the end of each time the final yield of silica were recorded.

Variation of concentration of sodium hydroxide on the yield of silica from samples

The concentrations of sodium hydroxide were then varied using 1.0, 2.0, 3.0, 4.0 and 5.0M. After each experiment, the yield of silica was determined and recorded.

Variation of reaction temperature on the yield of silica oxide from samples

The procedure was repeated by fixing the NaOH concentration that gave the optimum yields and fixing the reaction time at 1 hour. The reaction temperature was then varied using 80, 90, 100, 110 and 120°C.

Variation of reaction time on the yield of silica from samples

The reaction temperatures and sodium hydroxide concentrations were fixed at optimum values while the reaction time was then varied using 1,2,3,4 and 5 hours.

Characterization

Ashes produced at optimum ashing temperatures from both sugar cane bagasse and rice husk and the samples of silica obtained from them at optimum parameters were characterized using X-ray fluorescence (XRF) Philips X-ray spectrometer (model PW 2404).

RESULTS AND DISCUSSION

Table 1: Variation of ashing temperature on the yield of silica oxide from samples

Ashing Temperature(°C)	Yield of silica oxide (g)	
	Bagasse	Rice Husk
500	1.591	1.225
550	1.729	1.264
600	1.825	1.339
650	2.483	1.583
700	1.756	2.569
750	1.367	1.164
800	1.291	1.123

As indicated from table 1, sugar cane bagasse was ashed at temperatures of 500, 550, 600, 650, 700, 750 and 800°C respectively and the yield of silica from these samples were 1.591, 1.729, 1.825, 2.483, 1.756, 1.367 and 1.291g respectively, it can be clearly seen from the results that the yield of silica increases from the ashing temperature of 500 - 650°C. The temperature of 650°C was the optimum ashing temperature for the sugar cane bagasse because this temperature has the highest yield of silica of 2.483 g. The rice husk was also ashed at temperatures of 500, 550, 600, 650, 700, 750 and 800°C and the yield of silica obtained were 1.225, 1.264, 1.338, 1.583, 2.569, 1.164 and 1.123g respectively. The yields of silica increased from 1.225 - 2.569g before declining from 2.569g - 1.123g. Therefore, the value of 2.569g was the optimum yield of rice husk obtained at ashing temperature of 700°C.

The result from table 2 reveals that the ashing time of sugar cane bagasse was varied from 1-5 hours and the yield of silica obtained increases from 2.056 - 2.483g before declining from 2.483 - 1.015g. This result therefore suggests that the value of 2.483g being the highest yield of silica obtained from the sample ashed at 2 hours was the optimum. Therefore, the optimum ashing time for sugar cane bagasse was 2 hours. The result also showed that when the ashing time of rice husk was also varied from 1-5 hours and the resultant yield of silica increases from 1.027 - 2.721 g after which the yield decreases from 2.721 - 1.107g. The highest yield of silica obtained was 2.721 g at a time of 3 hours. Therefore ashing time of 3 hours was then chosen as the optimum for rice husk. The decrease in yields at higher ashing temperature was as a result of formation of crystalline silica formed which is soluble at higher temperature than amorphous silica.

Table 2: Variation of ashing time on the yield of silica oxide from samples

Ashing time (hr)	Mass of silica yield (g)	
	Bagasse	Rice husk
1	2.056	1.027
2	2.483	1.583
3	1.909	2.721
4	1.218	2.089
5	1.016	1.107

Table 3: Variation of concentration of NaOH on the yield of silica from samples

Conc. of NaOH(M)	Mass of silica yield (g)	
	Bagasse	Rice Husk
1	2.483	2.721
2	2.929	3.844
3	3.793	6.875
4	5.236	6.704
5	5.211	6.689

As shown in table 3, the sugar cane bagasse ashed was reacted with sodium hydroxide of different concentration. The sodium hydroxide concentration used was varied from 1 - 5M with a step change of 1M. The yield of silica increases from 2.483 - 5.236 g after which it declines slightly from 5.236 - 5.201 g. The

value of 5.236 g corresponding to 4M of sodium hydroxide concentration was chosen as the optimum for sugar cane bagasse. Also from the same result, the yield of silica from rice husk ash increases from 2.721 - 6.875g after which the yield decreases slightly from 6.875-6.689g. The value of 6.875g corresponding to 3M sodium hydroxide concentration is the optimum for rice husk.

The experimental result of table 4 revealed that the reaction temperature of sugar cane bagasse was varied from 80 - 120°C and the yield of silica obtained increases from 1.815-5.236g after which the yield declined from 5.236-1.211 g as seen. From the result, the value of 5.236g was the highest yield of silica and this was obtained at a reaction temperature of 100°C. Therefore, the optimum reaction temperature for sugar cane bagasse was 100°C. The result also showed that the optimum yield from rice husk was 6.875g. This value was obtained at a reaction temperature of 100°C. The temperature of 100°C was considered as the optimum reaction temperature for rice husk. The optimum reaction temperature for both wastes coincides showing that amorphous silica of plant origin can be dissolved at 100°C.

Table 6 shows the yields of silica from both wastes at optimum parameters. 30g of both sugar cane bagasse ash and rice husk ash produced 11.212g and 19.901g respectively at optimum production parameters.

Table 4: Variation of reaction temperature on the yield of silica from samples

Reaction Temp.(°C)	Yield of silica (g)	
	Bagasse	Rice husk
80	1.815	4.825
90	3.072	5.359
100	5.236	6.875
110	1.782	3.464
120	1.211	2.515

Table 5: Variation of reaction time on the yield of silica from samples

Reaction time (hr)	Yield of silica (g)	
	Bagasse	Rice husk
1	5.236	6.875
2	6.527	7.231
3	6.217	7.728
4	6.104	6.987
5	6.011	6.421

Table 6: The Yield of silica at optimum parameters from 30g each of sugar cane bagasse ash and rice husk ash

Parameters	Sugar cane Bagasse	Rice Husk
Ashing temperature	650°C	700°C
Ashing time	2hr	3hr
NaOH concentration	4M	3M
Reaction temperature	100°C	100°C
Reaction time	2hr	3hr
Yield	11.212g	19.901g

The characterization of sugar cane bagasse ash, rice husk ash and the final silica obtained from both ashes at optimum conditions were done using X-ray fluorescence (XRF). The result of the characterization in table 7 indicated that sugarcane bagasse ash contains 64.01 % silica (SiO₂) while rice husk ash contains 91.02 % silica. The final silica precipitated from the sugar cane bagasse ash contains 99.03 % silica while that from rice husk ash was 99.15 %. Minute quantities of K₂O and CaO were detected as impurities in the final product while few others are below the limit of quantification or not detectable (Nd). These results showed that significant quantity amorphous silica can be successfully produced from rice husk and sugar cane bagasse.

Table 7: Chemical compositions of sugar cane bagasse ash, rice husk ash and final silica samples

Compositions	Rice husk		Sugar cane bagasse	
	Ash (%)	silica (%)	Ash (%)	silica (%)
SiO ₂	91.02	99.15	64.01	99.03
K ₂ O	4.52	0.021	14.15	0.42
CaO	0.67	0.01	9.01	0.02
P ₂ O ₅	-	-	5.11	Nd
MgO	-	-	1.98	Nd
SO ₃	0.18	-	1.47	Nd
Al ₂ O ₃	1.98	-	1.51	Nd
Fe ₂ O ₃	0.31	Nd	1.55	-
MnO	0.06	Nd	0.31	-
Rb ₂ O	-	-	0.32	-
ZnO	0.03	Nd	-	-
CuO	0.09	Nd	0.02	-
Na ₂ O	-	-	0.14	-
SrO	-	-	0.01	-
TiO ₂	-	-	0.02	-
Cl ₂	-	-	0.31	-
Cr ₂ O ₃	-	-	0.03	-

Conclusion

The findings of this research work showed that amorphous silica can be successfully synthesized from sugar cane bagasse and rice husk. The optimum parameters for both sugar cane bagasse and rice husk respectively are: ashing temperature of 650°C and 700 °C, ashing time of 2hours and 3 hours, NaOH concentration of 4M and 3M, Reaction temperature of 100 °C both, Reaction time of 2hours and 3hours. The production at optimum parameters using 30g of each ash signifies that rice husk ash produces the highest yield of 66.3% compared to that of sugar cane bagasse with 37.4%.

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