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Modification of Silica Obtained from Rice Husk with 3-Aminopropyltriethoxysilane (APTES) and Use for the Extraction and Quantification of Toxic Metals from Tannery Waste Water Effluents

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Abstract

This study produces silica from rice husk and modified with 3-aminopropyltriethoxysilane (APTES) which can be employed for extractions and quantification of toxic metals from tannery wastewater effluents. The process results in the purification of water that can be recycled in industrial processes. Mixture of methods like X-ray diffraction, Scanning electron microscopy and infrared spectral methods were utilized to classified silica produced from rice husk samples from Kura, Kano State, Nigeria. The results obtained from the sample by XRF technique, shows many abundant oxides such as SiO_2 and AL_2O_3. The silica (SiO_2), Alumina (Al_2O_3), iron (Fe_2O_3), potassium (K_2O) and (MgO) contents of rice husk sample from Kura (68.3wt%, 0.477wt%, 0.047wt%, 0.021wt% and 0.077wt% respectively). From the FTIR spectra the samples show the presence of OH groups of silanol (Si-OH) and siloxanes (Si-O-Si-OH) is indicated in the 3452 cm^{-1} band, the intense absorption in 1096 cm-1, indorsed to the changes of asymmetric Si-O-Si stretch. The band at 798 cm^{-1} is caused by symmetrical stretching Si-O-Si changes, while the band at 457 cm^{-1} is caused by Si-O connections. The basal spacing of sample determined by XRD analysis is confirmed by a broad peak at around $2\theta = 30.3°$, indicating that the silica matrix are partially amorphous.and Braggs reflections. The formulated powder has peaks in 2θ regions of 26^0, 31^0,45^0, 56^0, 66^0, and 78^0. SEM image which has a layered structure.

Keywords: Rice husk, FTIR, XRF, XRD, and SEM;

1. INTRODUCTION

For the past decades rice as a cereal crop gain an extreme concern in Agricultural production in the world (Zhang *et al.,* 2012). It is term as a serial of grass species *Oryza sativa or Oryza glaberrima* that are highly consume in Asia and Africa continents. Similarly, rice became 3rd largest in the world production with the total of 741.5 million tonnes in 2014 (FAOSTAT 2018). It is argued that about 20 percent of paddy weight is source from its huks that was made from opaline silica and lignin (Noor Syuhadah, and Rohasliney 2012). Rice husk is term as waste that produce almost 545 million tonnes annually from agricultural production and it generates about 1/5 world rice production (Kenes *et al.,* 2012). Rice husk, can be transformed to produce high value silicon (Si) (Jung *et al.*, 2013), silica (SiO_2) (Liou and Yang 2011), silicon carbide (SiC) (Krishnarao and Godkhindi 2012), silicon nitride (Si_3N_4) and graphene (G) (Muramatsu *et al.,* 2014). Rice husk ash is generated from the burning or combustion process of Rice husk. It is important to reveal that the rice husk is a rise with the carbon content, utilized as a soil amendment, increase substance in cement and steel (Apoorv and Khalid 2014). Biochar produced by the thermal decomposition of the rise husk in the event of low supply of oxygen and temperature of less than 700^{0}C. The carbonized rice husk is caused by incomplete burning and can be utilized as a soil amendment and activated carbon (Apoorv and Khalid 2014).

Similarly, Silicon dioxide, also known as silica SiO_2, usually found as an inorganic material quartz and in many living organism (Fernandez *et al.*, 2015). It is among the most composite and abundant materials as a compound of numerous minerals and as synthetic product, including fused quartz, fumed silica, silica gel and aerogels. It is utilized as ingredients of micro electrons in the production of food and pharmaceutical industries (Lopez *et al.*, 2013). Hence, Water pollution arises as a result of waste products flown in into water, alter the water quality that affect human health and environmental settings. Water is an essential natural resource that is utilize for the entire human development (Bibi *et al.*, 2016). Healthy water for human use is necessary for good life in the world. Based on the report by world health organization (WHO) almost 80 percent of diseases are water borne. Therefore, in several nations of the world drinking water has not acquired WHO standards (Khan *et al.*, 2013). It is stressed that about 3.1 percent of deaths were as a consequence of unsafe water (Pawari and Gawande 2015). In this development, the release of toxc, industrial and domestic waste that flown through water ways including leaks, dams and rivers have become the major cause of harm to human health. Moreover, this pollution cause several diseases outbreak such as immune suppression, cholera, vomiting, reproduction failure, typhoid fever, skin and kidney disease (Khan and Ghouri 2011). In this regard, Tannery wastewater is considered to be one of the most polluting effluents due to large variety of toxic metals that range from chromium (Cr), Cobalt (Co), Lead (Pb), cadmium (Cd), Nickel (Ni), Selenium (Se) to Arsenic (As). Pollutant water came from tanneries has large amount of bad elements from hides and skins or as a result adding chemicals during the industrial process, that include sulfonated polyphenols, heavy metals, soluble carbohydrates and proteins (Oda and Nakagawa 2003).

Nonetheless, the peculiar features of silica formed from rice husk ash and adsorption properties, have accounted for their demand for different industrial applications such as its use for surface modification, binders, fillers for paper, pharmaceutical products, absorbent, adsorbent, catalyst, etc. Several studies has been done on the quantification and extraction of silica produced from rice husk analysis and characterization using different approaches such as infrared spectroscopy (FTIR), X-ray fluorescence (XRF), X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermo grametric analysis (TGA). Therefore, this study demonstrates the use of some of these techniques for characterization of silica produced from rice husk modified with APTES for the purification of tannery wastewater effluents.

2. METHODOLOGY

Collection of Rice husk

The paddy seed samples were collected from Kura rice mills industries (Kano state, Nigeria) and their husks were removed. All the glassware and plastic bottles were cleaned with soaps, washed with distilled water and drenched in 10% HNO_3 for 24 hours. They were cleaned with deionized water and dried in an oven at 80^0c for 24 hours. All chemicals reagents that were utilize in this study were of analytical grade purity and were used without further purification. Distilled water and deionized water would be applied for all synthesis and treatment processes. Hydrochloric (37%), sulphuric (90%), nitric (90%) acids, oxalic acid were all purchased from suppliers. 3-aminopropyltriethoxysilane (APTES) (99%).

Experiment

Silica was removed from rice husk ash familiarizing the technique of (Selvakumar et al., 2014). The obtained residue (RHA) was dissolved in 60 ml portions of 1N NaOH heated in a covered 250 ml Erlenmeyer containers for one hour to melt the silica and form a sodium silicate. The results was strained by What man No. 41 ashless filter paper, and the carbon remains were cleaned with 100 ml of hot water. The filtrates and washings were given time to cool to room temperature and were titrated with 1N HCl with constant stirring to pH 7. The gels of Silica commence when pH declined to less than ten and gets took time up to eighteen hours. Additionally, deionize water of one hundred ml was increase to gels which make them to break and turns to a slurry. Hence, the slurries are centrifuge for almost fifteen minutes at 2500 rpm in which the cleaning step keep repeated. Similarly, the gels were turn to beaker and dried in over at 80^oC for twelve hours to come up with xerogel.

Descriptions for the samples

X-ray fluorescence examination: The chemical structures of the silica αproduced from rice husk were done using X-ray fluorescence examination (X-supreme 8000) instrument.

Fourier transform infrared spectroscopy examination**:** FTIR examination was done using Cary sixty hundred and thirty equipment at Chemistry Lab Bayero University Kano, Nigeria. Entire FT-IR spectra were estimated between the ranges of 4500- 500 cm^{-1} at a resolution of 4 cm^{-1}.

X-ray diffraction examination: Bragg angular zone was explored by X-ray diffraction (XRD) model Bruker D2 phases, Analyzer 40 kV and 40 mA, cu Kα radiation source. The diffraction angle 2θ was scanned from 10^0 to 80^0 at scanning rate of 2^0 / min and step size of 0.05 ^{0} . In order to calculate the distance between the silicate layers, Bragg's law was used.

Scanning electron microscopy (SEM): Scanning electron microscope (SEM) (Phenom proxy, PW 100-004, magnification at 50x, Accelerating Voltage 10KV) was used to evaluate the surface morphology of the silica produced from rice husk sample.

3. RESULT

Classification of the silica formed from rice husk sample

Fourier transform infrared spectroscopy analysis (FTIR):

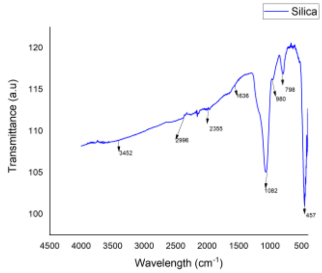

Figure 1: Fourier convert infrared spectra of silica sourced by RHA

The stretching vibration of the hydroxyl (-OH) group of silanol (Si-OH) in the silica and absorbed water molecules on the silica's surface are responsible for the broad band centred around 3452 cm^{-1}. A week as well as shoulder band cantered at nine hundred and eighty cm^{-1} supports the existence of a Si-OH bond, and an absorption band cantered at around 1636 cm-1 supports the occurrence of adsorbed water molecules that were as a result of O-H bond. 3 distinctive bands, linked to various modes of the bond, indicate the Si-O-Si operational framework. The presence of organic impurities originating from the husk used in the process is most likely responsible for the emergence of these organic related functional groups. (Wasinton et.,al, 2016).

XRF Analysis of silica produced from rice husk

S/N	Component	Mass%
1	SiO_2	68.3%
2	Al_2O_3	0.477%
3	Fe_2O_3	0.047%
4	T_1O_2	0.032%
5	P_{205}	0.09%
6	Na_2O	0.566%
7	K_2O	0.021%
8	MgO	0.077%

Table 1: Chemical properties, oxide composition (%) of Silica

According to the results of the tests, Si has the highest percentage of oxides with 68.8 percent, with x-ray diffraction examination revealing the existence of SiO_2 as an amorphous silica. (Ettah et.,al 2018)

XRD Analysis of silica created from rice husk

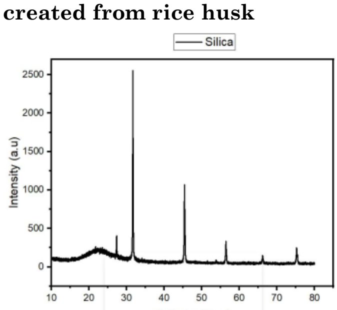

Figure 2: XRD analysis of silica produced from rice husk ash

Fig 2: shows an XRD pattern with distinct diffraction broad peak at around 2θ = 30.3°, indicating that the silica matrix are partially amorphous.and Braggs reflections. The formulated powder has peaks in 2θ regions of 26^0, 31^0,45^0, 56^0, 66^0, and 78^0.The purity of the silica nanoparticles is represented by the peaks. These Braggs reflections clearly indicated the presence of sets of lattice planes, and they can be indexed as face-centered-cubic (FCC) structure of silica nanoparticles formed in this synthesis are crystalline in nature (Manali et.,al 2020). As shown by the reflection at around 22.6^0 amorphous silica exists in RHA as a result of reflection refinement, whereas the crystalline form was obtained after composition formation (Fatimah et.,al 2014). Based on the specific reflection, it is concluded that the synthesis produces both anatase and rutile phase.

SEM Analysis of silica

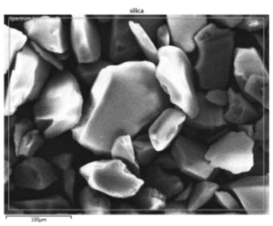

Figure 3: SEM images of silica obtained from rice husk

The morphological properties of the produced neat silica from rice husk ash was examined by using macroscopic techniques. Fig 3 represents SEM images of silica which has a layered structure. As Si ratio decreased, aggregation was diminished, and more homogeneous structure was observed.(Wu et.,al 2015) Irregular sizes and shapes, smooth surface compared to RHA structure with is non porous. (Hua et.,al 2016).

4. CONCLUSION

Silica produced from rice husk ash was successfully produced by sol-gel technique using NaOH to liquefy the silica and yield a sodium silicate solution which was dried at high temperature and was further modified with APTES as adsorbent for the removal of toxic metals from tannery wastewater effluents. The FTIR, XRF, XRD and SEM results of the synthesized silica confirmed the intercalation of the surfactant on the surface of the silica. Appearance of bands; indicate the Si-O-Si bond in the operational context of the siloxane in silica, namely the band centred at 1082 cm-1, the band centred at 798 cm-1, and the band centred 457 cm-1. XRF result revealed that the most abundant oxides in the sample are SiO_2 (68.3%) and $AL2O_3$ (0.477%) these results were found to be similar to those of previous studies. The morphology, chemical composition and basal spacing of silica were investigated by SEM, XRF and XRD examination.

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