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Preparation and Characterization of 'Green' Nano Silica from Rice Husks

Ityokyaa Daniel Terkula * Prof. R. A. Wuana Dr. M. S. Iorungwa
Department of Chemistry ,Federal University of Agriculture Makurdi *P.M.B.* 2373 *Makurd, Benue state, Nigeria*

Abstract
Silica with Nanostructure is a high quality silica that is used in many industrial areas. The study was aimed at preparation and characterization of nanao silica from rice husk by precipitation method. The rice husk ash (RHA) was obtained by washing and burning rice husk (RH) at 700^{o}C for 2 h in a muffle furnace. The RHA was subjected to Energy dispersive x-ray fluorescence (EDXRF) and silicon content was as high as 36.53 %. Indicating that there was silicon dioxide (silica) in RHA. Rice husk pure silica (RHS) was obtained by alkaline extraction method of 10g of RHA using 2.5 M sodium hydroxide. 9.3 g of RHS was obtained. The FT-IR spectral data which showed Absorption signals at 1069.7 cm^{-1}, 957.97 cm^{-1}, 797.7 cm^{-1} , 1632.6 cm^{-1} ,2885.0 – 3900 cm^{-1} and 2158.17 cm^{-1} , supported that the substance produced is silica. The RHS was subjected to precipitation by refluxing silica with 6 M HCl and dissolving the substrate in 2.5 M NaOH by continuous stirring for 1 hour on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5in order to produce nanosilica. The prepared Rice husk nano silica (RHNS) showed an X-ray diffractogram that has a strong broad peak at 18˚ to 36.5˚(2θ), which the absence of sharp peak indicates that nanosilica prepared is amorphous. FT-IR of RHNS support that the substance is silica. The SEM of the RHNS showed aggregates of silica forming fine globules of varied sizes. The EDXRF shows a complete removal of K from the RHA by alkaline treatment.
Keywords: Rice husk, precipitation, EDXRF, FT-IR, SEM ,Rice husk nano silica

1. Introduction

Nano Silica has wide application in our present day society. Its application ranges from it being a raw material for production of valuable materials like; cement, glass, porcelain, refractory, filler (for plastic, rubber and tire), vegetable oil refining, pharmaceutical products, detergents, adhesives, chromatograph column packing and Ceramics. It is also used as a precursor for preparing groundbreaking valuable materials like; SiC, Si3N4, elemental Si and Mg_2Si (CITATION Far06 \l 1033 Tzong-Horng,2004; Farook *et al.*,2006 ; CITATION Sin08 \l 1033 Singh *et al*.,2008 ; Pallavi *et al*., 2012; Yuvakkumara *et al*., 2012; Majid *et al.,* 2014; Nur *et al.,* 2015). Apart from the aforementioned application of Fine Silica powder (Nano silica), it is also finds uses in electronics, as substrates , insulators and sensors CITATION Eva14 \l 1033 (Eva *et al.*, 2014) . Despite the numerous uses of Silica in present day society, its preparation from nature tends to be expensive (Tzong-Horng, 2004; Pallavi et al., 2012; Yuvakkumara et al., 2012; Nur et al., 2015). Hence there is need to explore a cheap way to prepare this Nano Silica, given the dwindling nature of the Nation's economy.

This is where the use of rice husk (RH) appears to be relevant. Rice husk (RH), which is a by-product of the multistage processing of Rice. RH is an excellent source of high-grade Silica (Farook *et al.,* 2006; Majid *et al*.,2014). It can be used to prepare both amorphous and crystalline silica depending on the temperature of incineration (Iyenagbe and Othman, 2012). Silica that is prepared from RH is widely used in because of its amorphous nature, as the amorphous SiO_2 is more reactive when compared to crystalline SiO_2 (Farook *et al*.,2006).

The precursor for SiO_2 production is sodium silicate and it is currently produced by heating (melting) quartz sand with sodium carbonate(Na_2CO_3) at a temperature that is very high (1,300 ^{o}C). This method yields over 60 % SiO_2 and it is a very high energy method for the production of Silicates and SiO2 CITATION Pal12 \l 1033 (Pallavi *et al*., 2012) . However the amorphous nature of rice husk ash (RHA) SiO2 makes it extractable at low temperature and hence provides an alternative low energy method as compared to the current high energy method which involve heating (smelting) quartz sand with sodium trioxocarbonate (iv) at a great temperature of 1,300 ^{o}C CITATION Pal12 \l 1033 (Pallavi *et al.*, 2012) .

Rice is cultivated on a large scale throughout the world. Its cultivation generates rice husk on an annual basis, which most at times is regarded as waste in the world (Farook *et al.,* 2006; Singh *et al.,* 2008; Ezzat *et al*., 2012; Majid *et al*., 2014).

Rice husk is in abundant quantity in Rice producing countries such as India, Nigeria among others (Farook *et al*.,2006; Singh *et al.,*2008; Ezzat *et al*.,2012). Reports have it that for every ton of Rice produced, an approximate value of 0.23 tons of rice Husk is formed (Farook *et al.,* 2006). Despite the huge nature of rice husk, only a small portion of it is used as fuel. This creates problem of the disposal of the amount that is increasing year in year out (Farook et al., 2006; Singh *et al*., 2008; Ezzat *et al.,* 2012; Majid *et al*., 2014).

In addition to the disposal problem (as Rice Husk is littered everywhere often in the Rice mills), most

times the rice husk is burnt (without control) and it leads to release of smoke that is choking which causes the 'black cloud' phenomenon and this affects visibility, human health and global climate by emitting particulate matters and other gaseous pollutants which normally last for days in the affected areas (Farook *et al*., 2006; Majid et al., 2014). In this regard, the conversion of the rice husk in to a valuable material is urgently needed to avoid and control environmental pollution.

In light of the foregoing, it becomes imperative to investigate on a green route for the preparation of Nano Silica using a simple chemical method like Precipitation method.

2.0 Experimental method

2.1 Materials

All reagents were of analytical grade. The chemicals used as provided without purification and included sodium hydroxide pellet (NaOH), hydrochloric acid (HCl) (35.5%, 1.730 gmL^{-3}), Ammonium hydroxide (NH_4OH.), distilled water (H_2O), and Sulphuric acid (H_2SO_4))(97 % ,1.835 gmL^{-3}).

The materials that were used for the study included; Erlenmeyer flask, Filter apparatus, Thermometer, Stop clock, Magnetic Stirrer, and others instruments used were: X-ray diffractometer (XRD)(MD-10, Radicon limited, Russia), Energy dispersive x-rayflorescence spectrometer (EDXRF)(Skyray Instrument model EDX3600B), Scanning electron microscope (SEM)(phenomenon prix model mve016477830),Fourier transform infrared spectrometer (FTIR)(Agilent technologies cary 630), pH Meter, Oven, muffle furnace among others.

2.2 Preparation of Rice Husk Ash (RHA)

Rice Husk was obtained from Engr Paul Tyoga Mills at Ikpayongo Market, in Gwer- East Local Government Area of Benue State, Nigeria and washed comprehensively with distilled water to eliminate any adhering impurities. Rice husk was washed and air-dried at room temperature and 100g was burnt at 700^oC for 2 h in a muffle furnace (Majid *et al., 2014*; Premaratne *et al.,* 2013; Nittaya and Apinon, 2008).The washing and ashing was done at Chemistry Laboratory, Benue State University Makurdi.

2.3 Determination of chemical composition of RHA byEDXRF

The RHA chemical composition was determined by Skyray Instrument (EDX3600B) X-ray fluorescence spectrometer which applies XRF technology to conduct fast and accurate analysis of complex composition. The system detected elements between Magnesium (Mg, Z=12) and Uranium (U, Z =92) with high resolution and fast analysis. The sample was pulverised to fine homogeneous size and then pelletized. The Initialization (calibration), using pure silver standard, Selection of the working curve(ORE) according to the sample ,Testing of the sample was carried out at 40.0kv and 350 QUOTE and the intensity and content RHA recorded in a test time of 100s . The analysis was done at the laboratory of NASENI centre of Excellence Nanotechnology and advanced Material , Akure Ondo State.

2.4 Preparation of Pure Silica from Rice Husk Ash

10grams of RHA sample was stirred in 80 ml distilled 2.5M sodium hydroxide solution. The RHA was boiled in 250 ml Erlenmeyer flask for 3 h. The 250 ml Erlenmeyer flask was covered with aluminum foil to prevent the complete evaporation of the mixture. The filtration of mixture in was carried out and the residue was also washed with 20 ml boiling water into the filtrate for complete extraction of sodium silicate which is in the filtrate. The filtrate was cooled down to room temperature. 2.5M H_2SO_4 was added until pH 2 was reached and NH_4OH was added to adjust pH to 8.5 and allowed cool to room temperature .The solution was filtered and the filtrate was then be dried at 120ºC for 18hours in an oven. The pure silica obtained was weighed on a weighing balance. This technique was adopted from Majid *et al.,(2014)*; Premaratne *et al.,* (2013); Nittaya and Apinon, (2008). The preparation of the pure silica was done at chemistry laboartory, Benue State University Makurdi.

2.5 Characterization of Pure Rice Husk Silica (RHS)

The received product (pure riche hush silica) was finally investigated by Fourier transform infrared. FTIR spectra of RHS was recorded FTIR spectra of RHS was recorded on a FTIR Spectrometer (Agilent technologies cary 630) by scanning the sample 16 times with wave number of 4000-650 cm^{-1} using trasmittance method. The analysis was done in Microlabortory of Amadu Bello University Zaria.

2.6 Preparation of Nano Silica

The 9.3g Pure Silica obtained was refluxed with 6 M HCl for 4hours and the substrate washed repetitively with distilled water to make it acid free. It was then be dissolved in 2.5M NaOH by continuous stirring for 1hour on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5. The precipitated Silica was washed repetitively in distilled water until the filtrate became completely alkali free. The product obtained was dried at 50^oC for 48 h in the oven. This procedure was adopted from Majid *et al.,(2014)*;

Premaratne *et al.,* (2013); Nittaya and Apinon, (2008) .

2.7 Characterization of Nano Silica

a.Scanning electron microscopy (SEM) of nano silica

The morphology of synthesized Silica was examined by Scanning Electron Microscope (SEM)(phenomenon prix model mve016477830) . The scanning electron microscope was operated at 15 kV. Selected areas of interest was focused and micrographs were taken at different magnification (Majid *et al., 2014*; Premaratne *et al.,* 2013; Nittaya and Apinon 2008).

b. Phase analysis by X-ray diffraction

X-ray diffraction (XRD) of RHNS was examined using X-ray minidiffractometer MD-10, CuKα radiation (Radicon limited, Russia) using an acceleration voltage of 25 kV and current of 400 μA. The diffraction angle was scanned from 10 QUOTE to 75 QUOTE 2θ, at a rate of 3.25 ^{o} /min. (Majid*et al., 2014*; Premaratne *et al.,* 2013; Nittaya and Apinon 2008).

c. Fourier transform spectroscopy of RHNS

FTIR spectra of RHNS was recorded on a FTIR Spectrometer (Agilent technologies cary 630) by scanning the sample 16 times with wave length of 4000-650cm^{-1} using trasmittance method. The analysis was done in Microlabortory of Amadu Bello University Zaria.

d. Energy dispersive x-ray fluorescence (EDXRF) of RHNS

The RHNS chemical composition was determined by Skyray Instrument (EDX3600B) X-ray fluorescence spectrometer which applies XRF technology to conduct fast and accurate analysis of complex composition. The system detected elements between Magnesium (Mg, Z=12) and Uranium (U, Z =92) with high resolution and fast analysis. The sample was pulverised to fine homogeneous size and then pelletized. The Initialization (calibration), using pure silver standard, Selection of the working curve(ORE) according to the sample ,Testing of the sample was carried out at 40.0 kv and 350 QUOTE and the intensity and content RHNS recorded in a test time of 100 s.

3.0 Results and Discussion

3.1 Composition of RHA

The RHA on anlysis by energy dispersive X-ray fluorescence(EDXRF) as represented in Table 1 showed aChemical composition that majorly silicon (36.53 %) by content. This is higher than the value (35.47%) reported by Premaratne*et al.,*(2013) who burnt at 700 ^{o}C. This could be in agreement with Iyenagbe and Othman, 2012 who reported very high silica (hence Silicon) content. According to them varying composition of rice husk exist.

3.2 Percentage Yield of Pure Silica from Rice Husk Ash

The rice husk ash (RHA) sample after being extracted by 2.5 M sodium hydroxide generated the yield of pure silica up to 90.3%. This is to say that concentration of sodium hydroxide could had strongly effect on the dissolution of silica from as-received rice husk and it also removed some impurities which were not dissolved from the main product. This is confirmed from EDXRF of RHNS which shows a complete removal of K from the RHNS.

1.3.2 Characterization of Pure Rice Husk Silica (RHS) by FTIR

The functional groups present in the pure rice husk silica (RHS) prepared can be identified in the FTIR spectra of the RHS thus;

i. Vibration at 1069.7cm^{-1}, 957.97cm^{-1} and 797.7cm^{-1} are typical of Si–O–Si (siloxane bonds) bands attributed to the asymmetric stretching, symmetric stretching and bending, respectively (Farshid; *et al.,*2015 and Majid *et al., 2014*). These (3) three peaks are the main indices of the silica substances, which represent the successful preparation of silica(Majid*et al., 2014*).
ii. The absorption band of 1632.6cm^{-1} is for H–O–H bending vibration in water (An, *et al.,* 2010).
iii. The spectra show a broad band around Broad band from 2885.0 - 3900cm^{-1} is due to silinol OH groups and adsorbed water (Majid *et al., 2014*).
iv. Absorption band at 2158.17cm^{-1} could be impurities or vibration modes of the gel.(Ragini, *et al ;*2014)

1.3.5 **Characterization of Nano Silica**

a. Scanning electron microscopy (SEM) of nano silica

The SEM of the RHNS showed aggregates of silica forming fine globules of varied sizes . This is in accordance to Kapur,1985 who stated that it is a generally accepted fact that silica which is formed by incinerating rice husk below 800°C is amorphous and forms aggregates of silica forming fine globules or platelets of varied sizes.

b. Phase analysis by X-ray diffraction

The x-ray diffraction pattern of the prepared RHNS show major reflections or peaks of crystalline quartz from ICSD powder diffraction files (PDF) occur at Bragg 2θ angles of 44°. It can be seen that no defined peaks

corresponding to these Bragg 2θ angles are found in Figure 8. A rather broad peak spanning 2θ which is characteristic of amorphous structures is observed (Ragini, *et al.,* 2014). That is to say diffractogram obtained from phase analysis by x-ray diffraction show that the RHNS is in amorphous state. These findings are in agreement with the work of Kapur , 1985 , studied the structural behaviour of silica over a temperature range of 400-1500^{o}C and reported that at combustion temperature above 900°C, the silica in rice husk ash consisted of cristobalite and a smallamount of tridymite .Thus, to obtain amorphous silica from rice husk, the processing temperature should not exceed 700^{o}C, as phase transition to the crystalline structure of crystobalite would soon follow, although no specific temperature has been reported for this transformation (Iyenagbe and Othman, 2012).

c. Fourier transform spectroscopy

I. Vibration at 1066.0cm^{-1}, 957.9cm^{-1} and 797. 7cm^{-1}are typical of Si–O–Si (siloxane bonds) bands attributed to the asymmetric stretching, symmetric stretching and bending, respectively (Farshid; *et al.,*2015 and Majid *et al., 2014*). These (3) three peaks are the main indices of the silica substances ,which represent the successful preparation of silica.

II. The absorption band for H–O–H bending vibration in water is at 1625.1 cm^{-1} (An, *et al.,* 2010).

III. The spectra show a broad band around Broad band from 3700 - 2887cm^{-1} is due to silinol OH groups and adsorbed water (Majid *et al., 2014*).

IV. Absorption band at 2325.9, 2169,2023.9,2076.1 and1908.4cm^{-1} could be impurities or vibration modes of the gel.(Ragini, *et al ;*2014). From the FTIR spectra of the RHS and RHNS it could be said that there have same functional groups binding

d. Energy dispersive x-ray spectroscopy (EDX)

According to EDXRF chemical analysis data, synthesized RHNS may not have high chemical purity, due to the presence of chemical impurities(elements) which may be arise from the chemicals reagents used or the RHA. The Si content is reduced from 36.5293% to23.5979% against that increase from 35.47% in the rice husk ash to 41.07% in the nano silica as reported by Premaratne*et al.,*(2013)**.**

4 Conclusion

Nanosilica was synthesized from Rice husk ash (RHA) efficiently and effectively and was characterized by various analytical techniques. The percentage yield of nanosilica synthesized from burnt RHA at 700 ºC was 90.3%.

SEM analysis data showed that the nanosilica particles from RHA were in the agglomerate form and the particle size was in the range of 50-70 nm. The particle shape was found to be uniform. X-ray diffractograms showed a strong broad peak at 22.14^{o} (2 QUOTE) indicating nanosilica synthesized from RHA was amorphous. FTIR data revealed the presence of hydrogen bonded silanol group and siloxane groups in silica. The preparation of RHNS from agricultural byproduct (Rice husk), as opposed to that of quartz, provided an environmentally friendly solution with technically acceptable and economically attractive value added product.

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Table 1: Energy Dispersive X-ray Fluoresces Analysis of Rice Husk Ash (RHA)

Element	Intensity	Content(%)
Mg	0.0011	1.1225
Al	0.0056	1.7255
Si	0.2991	36.5293
P	0.0283	1.3506
S	0.0075	0.5557
K	0.0284	2.3116
Ca	0.0091	0.1657
Ti	0.0001	0.0000
V	0.0001	0.0070
Cr	0.0001	0.0000
Mn	0.0023	0.1641
Co	0.0001	0.0007
Fe	0.0073	0.7752
Ni	0.0010	0.0599
Cu	0.0025	0.0700
Zn	0.0042	0.1437
As	0.0001	0.0000
Pb	0.0002	0.0048
W	0.0005	0.1484
Au	0.0000	0.0000
Ag	0.0000	0.0009
Rb	0.0070	0.0683
Mo	0.0021	0.2315
Cd	0.0000	0.0000
Sn	0.0067	1.2560
Sb	0.0093	1.1639

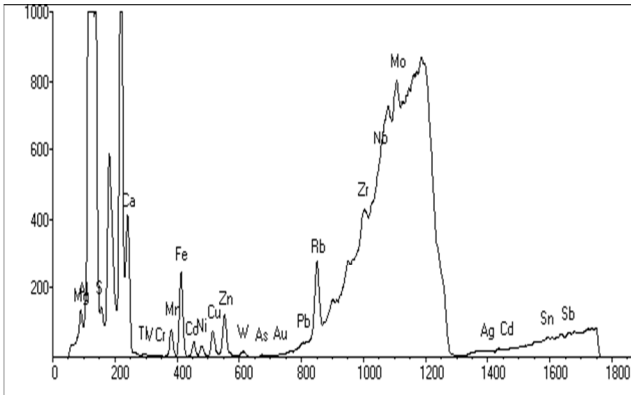


Figure 1: The Energy Dispersive X-ray Fluoresces Analysis of Rice Husk Ash (RHA)Spectrum

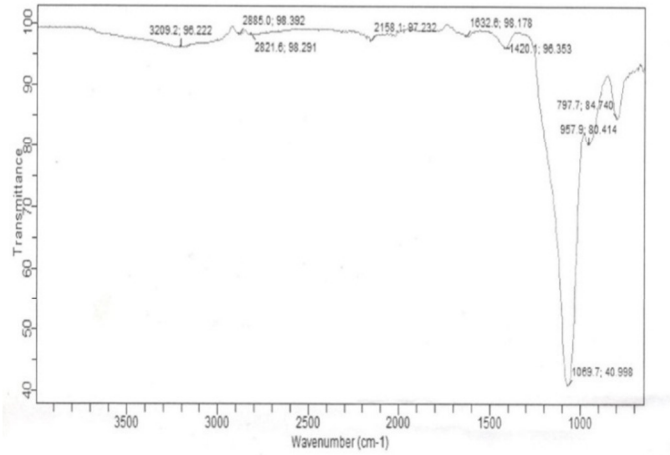
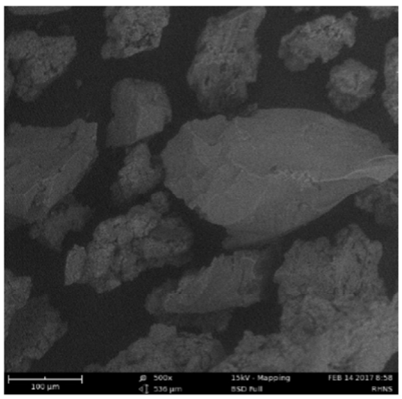
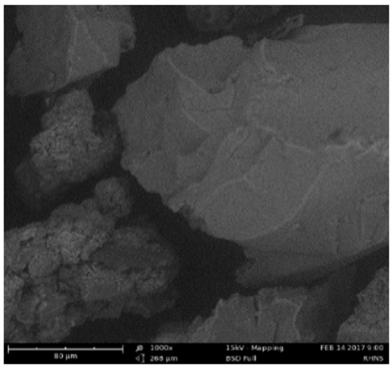


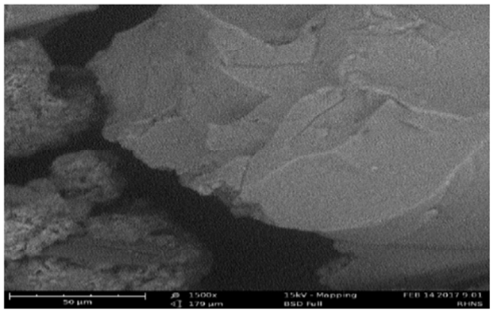
Figure 2: Fourier Transform Infrared(FT-IR) Spectra of pure Rice Husk Silica (RHS) Produced From Rice Husk Ash.



3a



3b



3c

Figure 3: SEM Micrographs of Prepared Rice Husk Nano Silica at Different Magnification

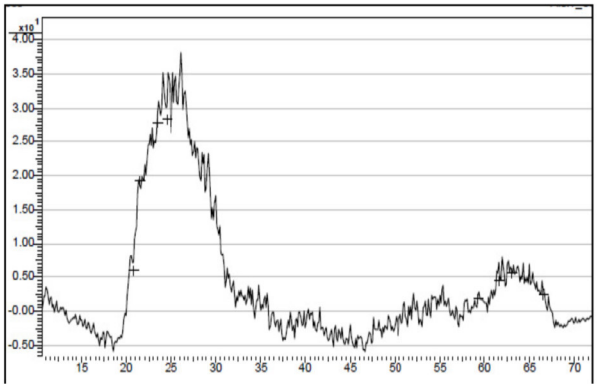


Figure 4: X-Ray Diffraction (XRD)Difractograms of Rice Husk Nano Silica(RHNS)

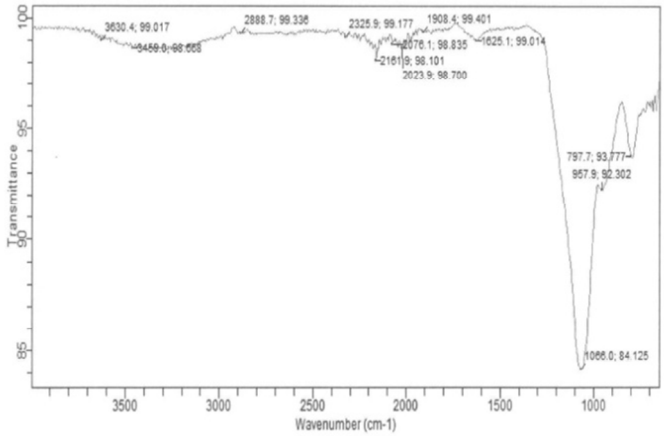


Figure5: Fourier Transform Infrared(FT-IR spectra of Rice Husk Nano Silica(RHNS)

Table 2: Energy Dispersive X-ray Fluoresces AnalysisRice Husk Nano Silica(RHNS)

Element	Intensity	Content(%)
Mg	0.0000	0.0000
Al	0.0038	1.2184
Si	0.2062	23.5979
P	0.0044	0.2066
S	0.0612	7.1369
K	0.0000	0.0000
Ca	0.0015	0.0187
Ti	0.0000	0.0000
V	0.0001	0.0069
Cr	0.0001	0.0007
Mn	0.0001	0.0000
Co	0.0001	0.0000
Fe	0.0029	0.3600
Ni	0.0012	0.0689
Cu	0.0027	0.0788
Zn	0.0052	0.1803
As	0.0001	0.0000
Pb	0.0000	0.0000
W	0.0007	0.2339
Au	0.0000	0.0000
Ag	0.0000	0.0000
Rb	0.0000	0.0000

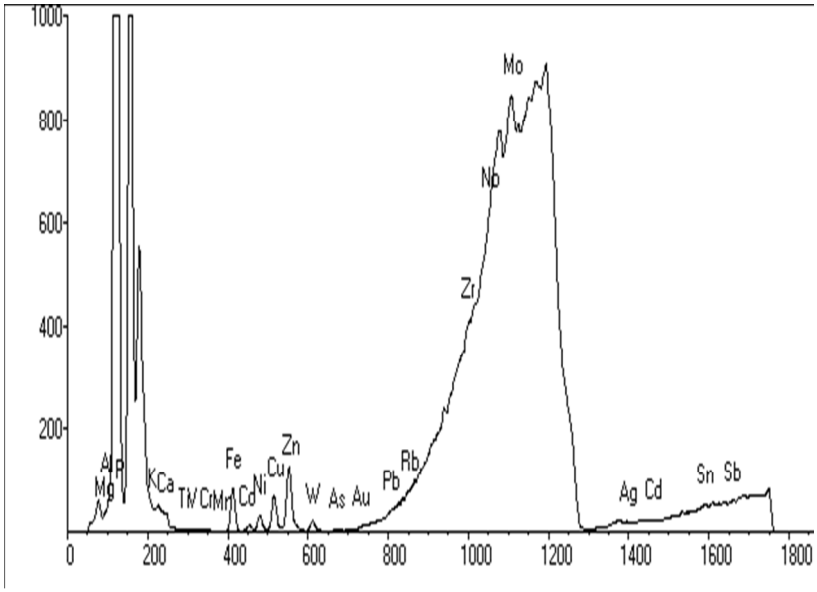


Figure6: Energy Dispersive X-ray Fluoresces AnalysisRice Husk Nano Silica(RHNS) spectrum