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EXTRACTION OF AMORPHOUS SILICA FROM WHEAT HUSK BY USING $KMnO_4$

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Abstract

Wheat husk an abundantly available agriculture waste in wheat growing areas, having an average of 9 percent of silica that can be retrieved as "Amorphous Silica" under controlled burning conditions. Extracting amorphous silica from wheat husk in commercial incinerators is difficult because of improper burning conditions and incomplete burning of inside heaps of husk, where availability of required oxygen can not be ensured. Potassium permanganate upon heating liberates oxygen and may provide the required oxygen to the husk during oxidation process. In present study the effect of potassium permanganate has been studied for extracting amorphous silica under controlled burning conditions. Wheat husk was treated with various dosages of potassium permanganate before subjecting to the tube furnace. The treated wheat husks were then heated at various temperatures. The ashes obtained were analyzed analytically and by XRD and FTIR. It has been observed from XRD patterns that $KMnO_4$ has helped in formation of amorphous silica where as FTIR spectra showed no carbon formation in treated samples where as analytical analyses confirmed better formation of amorphous silica.

Keywords: Wheat husk, Potassium permanganate, XRD, FTIR, Amorphous silica, Carbon.

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1. Introduction

Wheat husk (WH) is available in large quantities in wheat-growing areas. It has been considered as waste traditionally, but it can be put in to use in various chemical synthesis. Since the WH has significant quantities of silica, the recovery of this material is worth pursuing. The wheat husk when burned under controlled conditions can generate amorphous silica which has tremendous significance and good market value. It has been reported that 8 to 10 *per cent* amorphous silica can be recovered from WH [1]. The development of a commercial process that can achieve matching targets at a reduced cost is of interest. The chemical composition of the WH depends upon the type of fertilizer used, geographical conditions, and soil chemistry.

When the wheat husk is calcined under uncontrolled burning conditions, the ash formed may contain amorphous silica, crystalline silica, carbon residues and unburned husk. It has been reported that from rice husk and wheat husk the removal of carbon takes longer period once it is trapped during burning process and as a result recovery of amorphous silica become difficult [2]. Amorphous silica on the other hand has good market value which can be used in many chemical compounds [3].

It has been reported that amorphous silica can be produced by maintaining the combustion temperature below 500°C under oxidizing conditions for prolonged periods. Alternatively it can be produced at 590 °C with a holding time less than 1 min [4]. In commercial productions, generating the amorphous silica under controlled conditions is a difficult task because of other parameters. One of the difficulties is the uneven circulation of air in incinerators resulting in temperature gradients and ultimately formation of large portion of carbon and residues of husk. The low melting oxides of wheat husk gets fused with silica and form a glassy phase that prevents further burning [5]. Various incinerators with special features have been suggested in many designs for other types of husks such as rice husk [6,7]. Fluidized bed reactors are known to have excellent mixing and high reaction rates of gas-solid mixtures [8, 9, 10]

2. Materials and methods

Potassium permanganate is widely used in chemical industry to oxidize the chemicals. Potassium permanganate upon heating generates oxygen which facilitates the burning process of various types of husks [11]. In view of this characteristic the experimental study was conducted for the wheat husk by using dilute solutions of potassium permanganate. In order to evaluate the thermal degradation behaviors the wheat husks were soaked in various compositions of potassium permanganate and categorized as W1, W2 whereas untreated sample “W” was also prepared for comparison. The preparation procedure of each treated sample involved washing with de-ionized water followed by drying at 110^{o}C for 24 hrs. The dried samples were soaked in $KMnO_4$ solutions of 0.05, 0.005 normality for 30 minutes each and designated as W1 and W2

respectively. Each of these samples was again dried in an oven for 24 hours at 110 ^{o}C. Dried samples were then ground and stored in plastic bottles to protect from moisture and for further analyses.

2.1 Calcination process

Ground samples W, W1 and W2 were placed in four porcelain crucibles and subsequently calcined in (Carbolite) tube furnace which was programmed to rise in temperature at the rate of 10^{o}C/minute up to the desired temperatures. The residual weights and fixed carbon are given in Table 1.

Table 1: Average Residual Weights & Fixed Carbon in W, W1, W2

	Residual Weight %			Fixed Carbon %			
T(^{0}C)	(W)	(W1)	(W2)	Silica	(W)	(W1)	(W2)
300	51	48	42	9.0	42	39	33
400	19	18	17	9.0	10	6	4
500	18	15	13	9.0	2	1.5	0.5
600	15	14	12	9.0	3	0.4	0.3
700	13	12	11	9.0	0.3	0.2	0.1

2.2 XRD analyses

For XRD analysis the ground samples W, W1 and W2 were calcined up to 500^{o}C for 60 minutes and cooled at room temperature and were ground up to 200 mesh and dried at 110 ^{o}C for two hours before subjecting to XRD analyses.

The analyses were carried out in Philips Model PANalytical X-pert pro-machine. The Voltage of XRD machine with copper anode was set at 35 Kv, current intensity 35 mA and scanning spectrum at 5 to 70 degrees. The step size was adjusted to 0.050.

2.3 FTIR analyses

FT/IR JASCO model 4100 machine was used to obtain FTIR spectra. FTIR spectra were taken using potassium bromide pellet technique. Approximately 1 mg of materials W, W1 and W2 were mixed with 110 mg of KBr each. The mixture was ground, dried, compressed, and pellets were prepared to scan. The FTIR spectra of the samples were obtained in the range of 300 cm^{-1} to 4000 cm^{-1} and are shown in Figs. 4,5.

3. Results and discussions

Effect of potassium permanganate on residual weights and fixed carbon has been shown in Table 1. Silica was found approximately 9 *per cent* after loss on ignition (LOI). It is evident that in the range of 300^0C to 700^0C, $KMnO_4$ has successfully oxidized more carbon from samples W1, and W2 than from sample W. In case of sample W2 the residual weights and fixed carbon are found the lowest. The results in table 1 shows only the residual weights and fixed carbon formed after calcinations. The formation of amorphous silica can not be determined from the table1, to this end XRD results and analytical methods were used.

To find out the percentage of amorphous silica, rapid analytical technique has been used [12]. This technique involves the titration of $Ba(OH)_2$ solution against the solution of glycerol and WHA by using phenolphthalein as an indicator. The results are summarized in Table 2.

Table 2. Amorphous silica determined by rapid analytical method

Temp. ^{0}C	% Amorphous silica		
	W	W1	W2
500	85	88	90
600	60	65	73
700	51	55	61

The X-Ray diffraction patterns generated for the materials are shown in Figures 1-3. It may be observed that all diffused peaks at Theta = 22 degrees confirming the formation of amorphous silica in general. It has been reported that diffused broad peak at Theta = 22 degrees indicates amorphous silica along with some crystalline silica [13]. The small peak for the case of samples W and W1 indicates a higher quantity of crystalline silica (Figs 1,2). For the case of sample W2 broad peak is diffused at Theta = 22 degrees, without having any small peak, indicating the formation of more amorphous silica along with minor quantities of crystal silica. It may be

observed by comparative study of XRD results that dilute solution of potassium permanganate has helped in oxidation and formation of good amorphous silica.

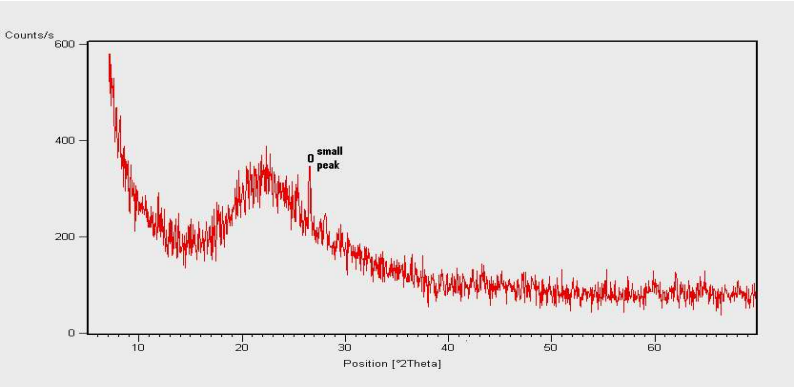

Fig 1: XRD of untreated wheat husk ash

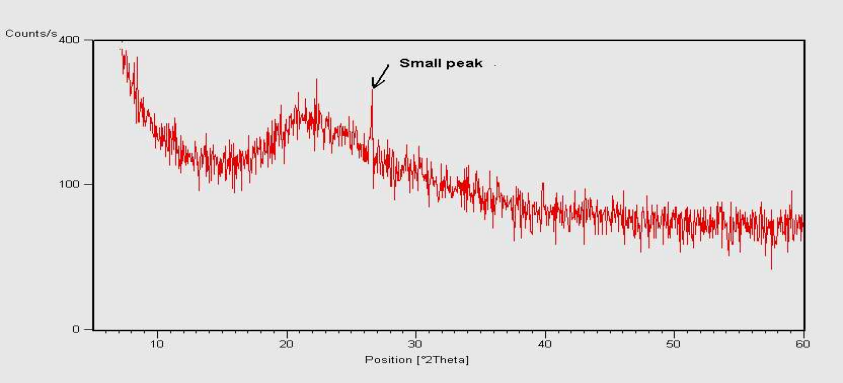

Fig 2: XRD of ash of treated wheat husk (0.05 N solution of **KMnO4)**

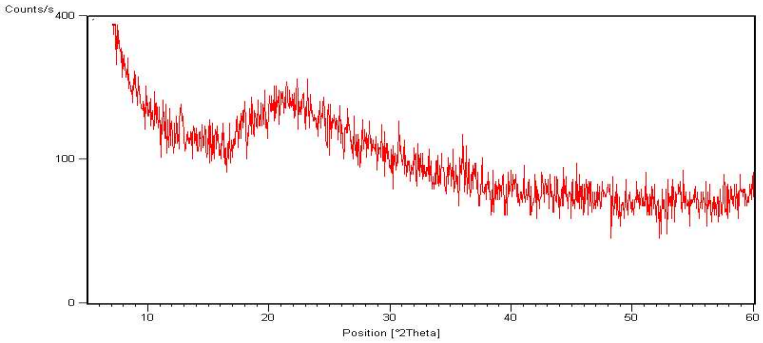

Fig 3: XRD of ash of treated wheat husk (0.005 N solution of **KMnO4)**

FTIR spectra of W and W2 have been taken and are shown in figures 4 and 5. Five bands of each sample (W and W2) can be observed with shifting of one band from 1640 cm^{-1} to 2260 cm^{-1} degree.

It may be observed that the bands 461 cm^{-1} to 476 cm^{-1} belong to O-Si-O bending vibration. Bands 1091 cm^{-1} to 1097 cm^{-1} and 797 to 806 cm^{-1} belong to O-Si-O stretching vibration. Bands at 3427 cm^{-1} to 3636 cm^{-1} indicate the chemisorbed water and surface OH groups. The intensity of peak of sample W is the highest that indicates more chemisorbed water. The bands from 1632 cm^{-1} to 1647 cm^{-1} belong to C-O group. The carbon band for W has been observed at 1640 cm^{-1} however for W2 no band has been observed indicating absence of carbon formation.

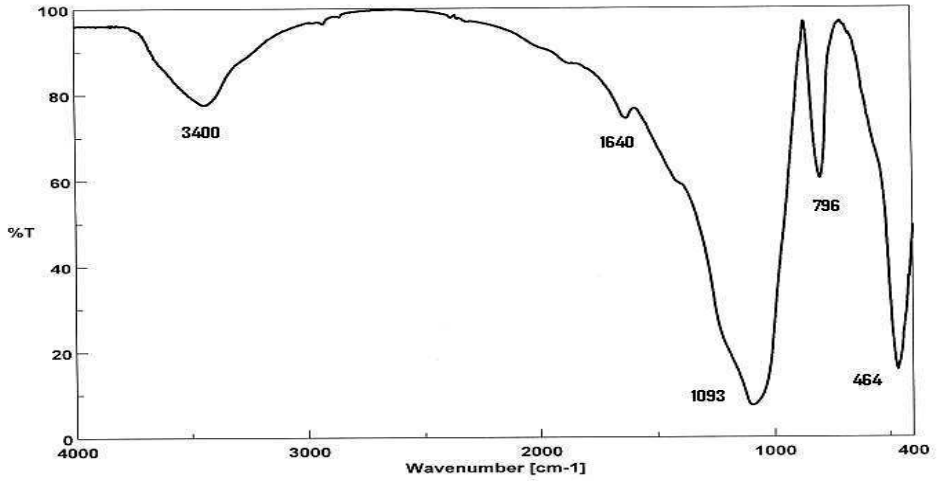

Fig 4: FTIR of sample W

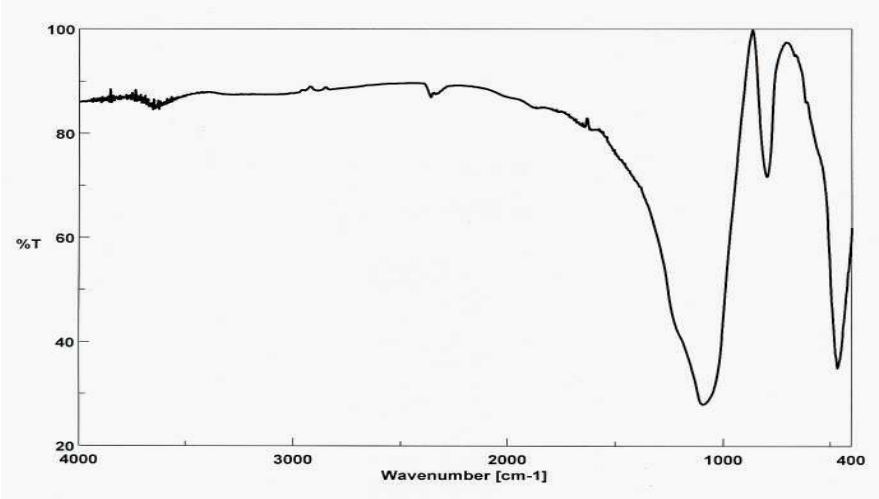

Fig 5: FTIR of sample W2

4. Conclusions

Wheat husks treated with various normal solutions of potassium permanganate has been studied for the formation of amorphous silica. Ashes obtained by treated and untreated wheat husks were analyzed analytically and by XRD and FTIR. Analytical results showed that ashes treated with dilute solutions of potassium permanganate render silica with higher percentage of amorphous silica. On the basis of the XRD analyses it has been observed that the oxidizing agent used has helped in formation of amorphous silica. Fixed carbon has been detected in FTIR spectra for W whereas no carbon has been detected in the case of W2. The optimum concentration of potassium permanganate was established as 0.005 N for wheat husk with calcinations temperature of 500°C.

Acknowledgement

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