Characterization of Wheat Hull and Wheat Hull Ash as a Potential Source of SiO_2

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Wheat is a very important agricultural product, and is among the grains that has the highest sustenance value in the world. Every year large amounts of wheat hull is produced which has a low economic value. The present study aimed to characterize the structure of wheat hull and wheat hull ash obtained after burning wheat hull at 400 to 1000 °C for 5 h. This study also investigated the potential of wheat hull as a source for the production of silica xerogel. X-ray diffraction patterns, scanning electron microscope micrographs, the chemical properties, the particle morphology, particle size, surface chemistry of wheat hull, wheat hull ash, and its silica xerogels were determined by the use of various instruments and analysis techniques. A temperature of 700 °C was found to be the optimum ashing temperature with maximum amorphous silica content. The BET surface area of ashes ranged from 7.21 to 0.11 m^2/g. The study demonstrated that silica xerogel powder with 93.9% silica content was obtained by the use of wheat hull after heat treating at 700 °C for 5 h.

Keywords: Adsorbent; Agricultural waste; Wheat hull; Wheat hull ash; Silica; Silica xerogel

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INTRODUCTION

A member of the triticale group of grains, wheat is one of the most important food crops in the world (Sahrawat *et al*. 2003). It is the world's second most commonly produced and consumed grain because of its high production rate, it is easy grow, and it can be converted into a variety of convenient foods, such as biscuits and cakes, pasta, noodles, and some breakfast cereal products (Sahrawat *et al.* 2003; Jones 2005). Thus, it has an important role in nourishment as it is the most important source of plant protein in the human diet and it supplies essential vitamins and minerals (Sahrawat *et al.* 2003; Jones 2005). In 2009, the world production of wheat was 656 million metric tons and approximately 138 million metric tons were produced by the European Union (FAOSTAT 2009). After the shelling process of the wheat, large quantities of wheat hull are generated as a waste material. Wheat hull is a lignocellulosic by-product which constitutes about 15–20% of wheat (Bledzki *et al.* 2010). Wheat hull contains approximately 6% protein, 2% ash, 20% cellulose, and 0.5% fat and non-starch polysaccharides (Hoseney 1994). Wheat hull is a low-cost, low density agricultural residue that is disposed of and left to decay. However, it is generally burned in open air fires (Kaya *et al.* 2008), producing wheat hull ash. The improper dumped wheat hull ash can damage land fertility due to its trace heavy metal content. Thus, wheat hull ash is referred to as the "waste product of food industry," and can cause environmental pollution. Only a small percentage of wheat hull is used as cattle food and fuel and the rest is usually taken to land fill areas. Wheat hull has been evaluated for use in biofuels production, such as biochar and bio-oil (Bledzki *et al.* 2010; Motojima *et al.* 1995). It has also been used to produce the fibers of Si_3N_4 by Motojima *et al.* (Zhang and Khatib 2008). Moreover, wheat hull ash was used as a concrete additive in the construction industry (Mani *et al.* 2004).

Many studies in the literature have focused on the use of rice hull ash, which is similar to wheat hull in that it has a high content of silicon, in various fields as a renewable source, as an adsorbent (Foo and Hameed 2009; Kim and Choi 1998; Ozgul-Yucel and Turkay 2003; Srivastava *et al.* 2008; Srivastava *et al.* 2009; Wang and Lin 2008a; Wang and Lin 2008b), as the raw material for silicon (Watari *et al.* 2006; Kalapthy *et al.* 2000; Kamath and Proctor 1998), and as a construction material (Nehdi *et al.* 2003). However, wheat hull has not been sufficiently utilized in industries. There have been few studies on the use of wheat hull for the production of silica-based compounds (Zhang and Khatib 2008; Terzioglu and Yucel 2012). Silica xerogel is one of the most important silica-based powders and is used as a carrier in drug delivery, drug eluting bone replacement, catalyst, and sorbent (Lee *et al.* 2010; Huang *et al.* 2003). The utilization of wheat hull ash in the various areas of industry will contribute both economically and also in waste management strategies. From the economic point of view, the utilization of sodium silicate, which is the precursor of silica-based products from ashes, is an alternative low-energy method when compared to conventional production that requires high energy because sodium silicate is obtained from the reaction of quartz sand with sodium carbonate at 1300 °C (Kalapathy *et al.* 2002 ; Kamath and Proctor 1998). Wheat hull will gain an additional value (Skoulou and Zabaniotou 2007). Thus, the present study presented a new application area for the efficient evaluation of this type of waste. In the literature, there are not any studies about the evaluation of amorphous silica of wheat hull ash for silica xerogel production. As a result, a novel utilization area for wheat hull and wheat hull ash containing a high proportion of silica was proposed in the present study. For this purpose, the investigation of the characteristics such as morphology, composition, and the surface chemistry of wheat hull, wheat hull ash samples, and its silica xerogel were carried out to evaluate the feasibility of using them as a source of silica-based materials production.

MATERIALS AND METHODS

Materials

The wheat hull sample was obtained from the Doruk Marmara Flour Factory located in the Marmara region of Turkey. Wheat hull ash was obtained by burning these hulls in the laboratory. The burning process was carried out by the use of an incineration ash furnace (Protherm). The chemicals used in the production of magnesium silicate, which were analytical reagent grade, were supplied by Merck (Darmstadt, Germany).

Dry Matter and Ash Content of Wheat Hull

Wheat hull was dried at 110 °C to a constant weight to determine the dry matter content.

$$\% \text{ dry matter} = (W_1 - W_2)/W_3 x 100 \qquad (1)$$

where W_1,W_2, and W_3 are the weight of the wheat hull after drying, tare of crucible, and original sample, respectively.

Then, 10 g of wheat hull were put into a ceramic crucible, and burnt at 550 °C for 12 h until the temperature decreased to 250 °C. The samples were taken from the crucible and then put in a desiccator to cool slowly (Nielsen 1993). The ash content was calculated as follows:

$$\%\ \text{ash (dry basis)} = (W_1 - W_2)/(W_3 \bullet W_4)x100 \tag{2}$$

where W_4 is the dry matter coefficient of wheat hull.

Characterization of Wheat Hull and Wheat Hull Ash

Ash samples were prepared by heating the wheat hull at temperatures of 400, 500, 600, 700, 800, 900, and 1000 °C for 5 h. The ashing time and temperature plays an important role on the chemical and physical properties of ash. As the ashing time is setted short (0-2 h), large amounts of unburnt carbon remains in the ash. However, the higher ashing temperatures and longer times cause crystalline silica formation (Della *et al.* 2002). When the ashing time was 5 h, the silica in wheat hull ash remained amorphous, and thus 5 h was chosen as the ashing time. Characterization of wheat hull and wheat hull ash samples was performed by using various analysis methods and equipments. Scanning Electron Microscope (SEM) micrographs of the samples were taken by the use of a Scanning Electron Microscope (JEOL JSM 5410 LV, Japan; CamScan- Oxford Instruments, France) and surface areas of the samples were determined by the use of a BET (Costech 1042 Sorptometer, Italy) instrument.

X-ray diffraction patterns of the samples were obtained with an X'Pert PRO PANalytical (Holland) XRD system using an acceleration voltage of 40 kV and a current of 45 mA. The diffraction angle was scanned from 5^o to 90^o 2θ, with a step size of 0.003.

Fourier transform infrared spectra of the samples were obtained with use of a SHIMADZU, IR Prestige 21; USA, in the range of 400 to 2000 cm^{-1}, processing at a resolution of 4 cm^{-1} with 64 scans.

The chemical composition of wheat hull and wheat hull ash was determined by the use of an Inductively Coupled Plazma (ICP) system (Perkin Elmer Optical Emission Spectrometer Optima 2100 DV, USA). Prior to the ICP analysis, the samples were prepared by adding 3 mL of HNO_3 (65%), and 1 mL of HF (40%) to 0.05 g of ash in a Teflon digestion vessel. A blind solution was prepared by the same method. All of the trials were conducted in parallel. The samples were then digested by the use of microwave equipment (BERGHOF SpeedwaveTM MWS-3, Germany) according to the following four-steps program: (i) 5 min at 170 °C and 50% power; (ii) 30 min at 200 °C and 80% power; (iii) 15 min at 200 °C and 80% power; (iv) 1 min at 100 °C and 10% power. The dissolved samples were diluted to a final volume of 25 mL with deionized water and put into plastic containers until the ICP readings. All of the samples were analysed in triplicate.

Silica Xerogel Production

The synthesis procedure of silica xerogel powder is given in Fig. 1. Silica xerogel was extracted from wheat hull ash burned at 700 °C for 5 h adapting the method of Kalapathy *et al.* (2000). The silicate solution was prepared by boiling 20 g of wheat hull ash with 250 mL of 1N NaOH solution in a covered 500 mL Erlenmeyer flask for 2 h with constant stirring as given in Equation 3. The mixture was then filtered under vacuum; the upper carbon residues were washed with 60 mL of hot water to separate the clear sodium silicate solution from carbon residues (Ozgul-Yucel *et al.* 2004). The silicate solution was allowed to cool to room temperature and was titrated with 1N HCl with constant stirring to a pH of nine. The soft gel was aged for 20 h. The obtained gel was gently broken and the slurry was centrifuged for 15 min at 2500 rpm. The clear supernatants were poured. The slurry was washed with deionized water several times and the centrifugation was repeated. The gel was dried in an oven at 80 °C for 12 h to produce silica xerogel.

$$SiO_2 + 2NaOH \longrightarrow Na_2SiO_3 + H_2O \qquad (3)$$

Characterization of Produced Silica Xerogel

The silica xerogel powders were characterized by XRD, XRF, and FTIR analysis techniques to identify its structural properties. The elemental content of the produced silica was roughly estimated with an X-ray fluorescence spectrometer (XRF, Panalytical Minapal 4, The Netherlands). The crystalline structure of the produced silica was examined by an X'Pert XRD (Philips Panalytical X'Pert Pro, The Netherlands) with CuKα radiation using an acceleration voltage of 45 kV and a current of 40 mA. The patterns were recorded for grounded samples in the FTIR spectra. The FTIR spectra of the samples were obtained in the range of 750 to 4000 cm^{-1} with the use of a Perkin Elmer FTIR spectrometer.

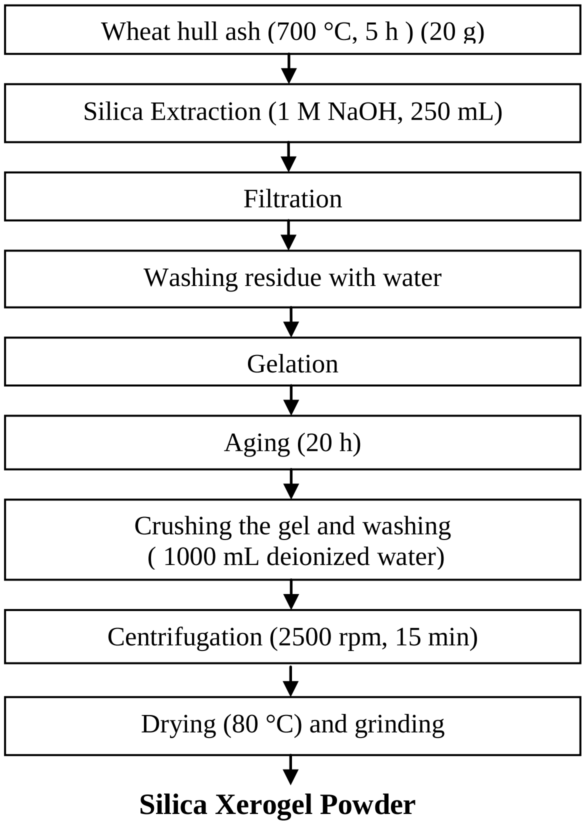

Fig. 1. Flow diagram of the procedure used to produce silica xerogels from wheat hull ash

RESULTS AND DISCUSSION

Wheat Hull Characterization

Physical and chemical characteristics of wheat hull

Dry matter content and the ash content, SEM micrographs, BET surface area, and FTIR spectrums of wheat hull were determined and presented.

The dry matter content of wheat hull was calculated with Equation 1 and found to be 7.7%. The ash content of wheat hull was calculated with Equation 2 and found to be 9.95%.

The identified functional groups from the FTIR spectrum of wheat hull are shown in Table 1. In our previous work, we found that the O-Si-O band in an FTIR spectrum of wheat hull appears at 1032 cm^{-1} (Terzioglu and Yucel 2012). The bending vibrations of H-OH can be seen at 1631 cm^{-1}. The peak observed at 920 cm^{-1} was assigned to Si–OH stretching vibration. The peak at about 1726 cm^{-1} was related to the C=O stretching of the uronic and acetyl ester groups of hemicellulose, or to the ester linkage of the carboxylic group of the ferulic and p-coumaric acids of lignin and/or hemicelluloses (Sun *et al.* 2005). The amide I band that indicates the presence of 80% of the C=O stretching of the amide group and C–N stretching modes was observed at around 1640 cm^{-1}. The peaks between 1450 and 1370 cm^{-1} corresponded to the C–H symmetric and asymmetric deformations. The characteristic peak of β glycosidic linkages between the sugar units was observed at 900 cm^{-1} (Bledzki *et al.* 2010).

Table 1. The functional groups of wheat hull determined by the FTIR analysis

Wave numbers (cm^{-1})	Functional groups
1726	C=O stretching
1640	C=O stretching
1631	H–OH bending
1450-1370	C–H symmetric, asymmetric
920	Si–OH stretching
~900	β glycosidic linkages

Surface properties of wheat hull

Figure 2 demonstrates the SEM (32 x -2000x) microstructural view of wheat hull. Some of the wheat hull fibers have a cornered and rounded shape, while others are long. Figure 2-C provides a 200x zoom view of a section in Fig. 2-A, where stalactite structures and diffused circular parts can be observed for the majority of the surface of the flat section. Semi-quantitative results (EDS) of the SEM equipment demonstrate that the stalactite parts have more K and Si elements than the diffused circular parts. Fig. 2-E provides a structure that looks like a corncob. It is known that the silica, which is found amorphous in the rice hull structure, is located on the bulges on the outer hull (Krishnarao *et al.* 2001). As Fig. 2-E gives a similar view, it is determined that silica is intense in this structure as the result of EDS analysis (Table 2). It has a fibrous structure besides the corncob structure.

The surface area of wheat hull was measured by the use of a surface area meter and the BET surface area of wheat hull was found to be 0.92 m^2/g. A small surface area of wheat hull without any thermal or chemical process is a normal finding.

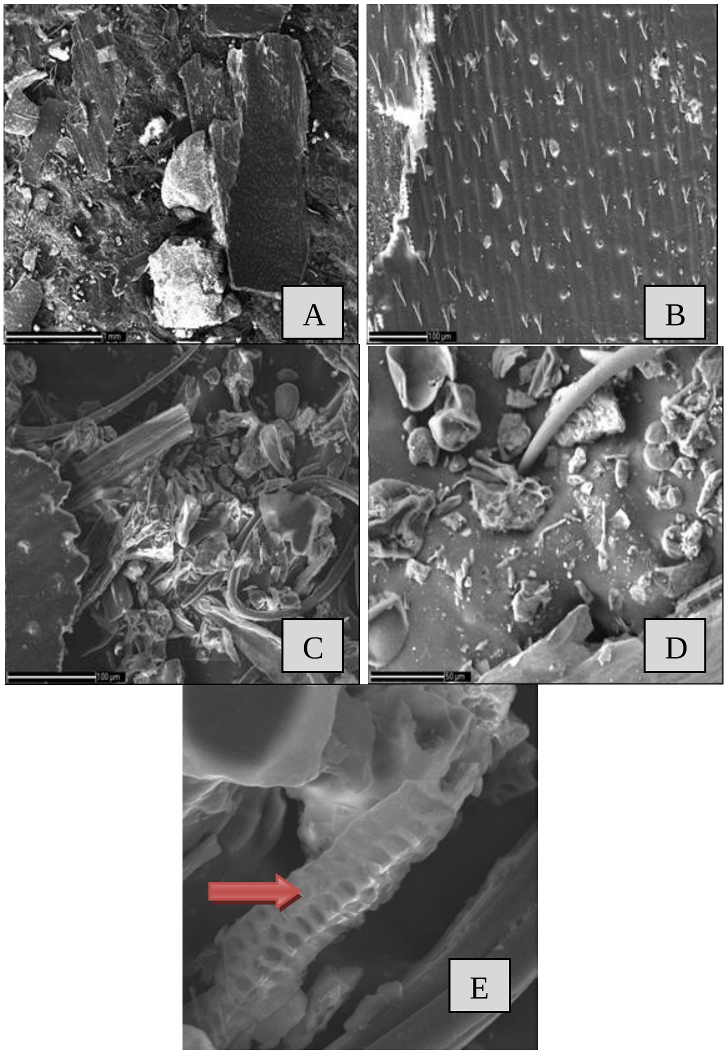

Fig. 2. The SEM micrograph of wheat hull A) 32x B) 200 x C) 300x D) 500x E) 2000x

Table 2. Chemical Composition for Corncob Structure (Fig.2-E) by use of EDS

Element	**Si**	**Na**	**Cl**
Atomic (%)	65.26	22.91	11.83

Wheat Hull Ash Characterization

The structure and composition of the wheat hull ash obtained at temperatures of 400, 500, 600, 700, 800, 900, and 1000 °C for 5 h was characterized to use as a source of silica production. All of the characterization results were presented in sections below.

Physical and chemical characteristics of wheat hull ash

Views of wheat hull ash, which was burned at different temperatures for 5 h, are given in Fig. 3. As can be seen in Fig. 3, the wheat hull ash burned at 400 °C and 500 °C has a light color while the color gets darker at 600-800 °C, and gets lighter again as the temperature goes as high as 900 °C and 1000 °C. At higher combustion temperatures, the carbon gets oxidized and burns off. Thus, the presence of unburnt black carbon decreases, and the colored oxides of metallic ingredients that are in the hull influences the color of ash, resulting in increasing lightness (Chandrasekhar *et al.* 2006).

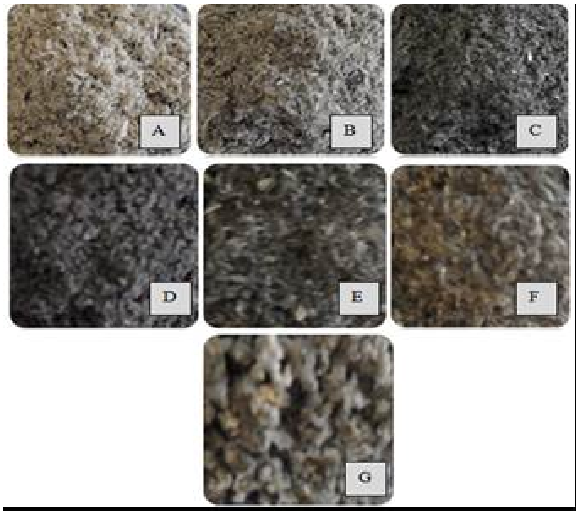

Fig. 3. Images of wheat hull ash burnt at various temperatures for 5 hours A) 400 °C B) 500 °C C) 600 °C D) 700 °C E) 800 °C F) 900 °C G) 1000 °C

The chemical compositions of wheat hull ash burned at various temperatures for 5 h are given in Table 3. The ashes were composed of SiO_2, K_2O, MgO, Fe_2O_3, Na_2O, Cr_2O_3, MnO_2, and CaO. The major component of the ashes was SiO_2. The other major components were found as K_2O and CaO. The remaining chemical composition is composed of the unburnt carbon, hydrated water, and other residues of ash. It was observed that as the temperature increased, the amount of silica in the wheat hull increased as well. Although the silica content is higher at 800-1000 °C, the working temperature was set at 600-700 °C because the XRD results and the studies in literature demonstrate that silica is converted into a cristobalite form at temperatures above 700 °C. However, it is easier to extract amorphous silica by the low energy method (Thuadaij and Nuntiya 2008). Furthermore, the ash does not pose dangers for human health when in amorphous structure, but it is deemed damaging for humans when it is in the crystal form since it is less reactive when it contains silicon dioxide (Ferrero 2010). In this study, 700 °C was chosen as the working temperature because the appearance of the cristobalite silica was so slight.

The FTIR spectrum of wheat hull ash after burning at 700 °C for 5 h is given in Fig. 4. The broad peak at 1040 cm^{-1} and the small peak at 799 cm^{-1} represent the Si-O-Si stretching modes.

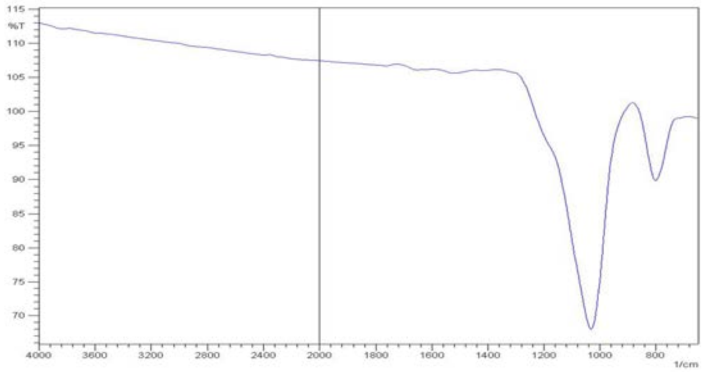

Fig. 4. FTIR spectrum of wheat hull ash

Table 3. Chemical Compositions of Wheat Hull Ashes Burned at Various Temperatures

	Wheat Hull	**400 °C**	**500 °C**	**600 °C**	**700 °C**	**800 °C**	**900 °C**	**1000 °C**
SiO_2(%)	11.04	38.36	37.72	43.22	49.17	58.15	58.86	58.75
K_2O(%)	1.25	10.81	12.88	11.30	11.27	11.86	11.12	12.50
MgO(%)	0.21	0.77	1.14	0.99	0.95	1.37	0.76	1.45
Fe_2O_3(%)	0.32	0.76	1.06	0.84	0.86	1.00	1.00	1.08
Na_2O(%)	0.09	0.12	0.17	0.16	0.27	0.72	0.40	0.62
Cr_2O_3(%)	0.0001	0.0003	0.0004	0.0004	0.0003	0.0005	0.0004	0.0005
MnO_2(%)	0.01	0.02	0.03	0.02	0.03	0.03	0.03	0.03
CaO(%)	2.43	4.08	6.42	5.46	5.46	7.24	4.61	6.14
Al_2O_3(%)	0.51	0.62	0.46	0.15	0.28	0.35	0.18	0.61
Total content(%)	15.86	55.54	59.88	62.14	68.29	80.72	76.96	81.18

XRD analysis of wheat hull ash

Figure 5 shows the X-Ray diffraction pattern images; these images help to determine the structural form of SiO_2 available in wheat hull ash burned at various temperatures. The silica found in the hull burned at 400 °C-600 °C had an almost completely amorphous structure (Terzioglu and Yucel 2012). At 700 °C, the cristobalite form begun to emerge, though slightly, along with the quartz structure. At 800-900 °C, the cristobalite structure increased to a certain extent. In Fig. 5-D, the XRD pattern shows a sharp peak at about $2\theta = 22°$ corresponding to a cristobalite structure. Therefore, the cristobalite is the more important crystalline phase in the ashobtained at 1000 °C. In a study on rice hull conducted by Proctor, it was observed that with the increase in temperature, the transition to the crystal form in the ash structure increases, and the existing surface area of the ash decreases (Proctor 1990).

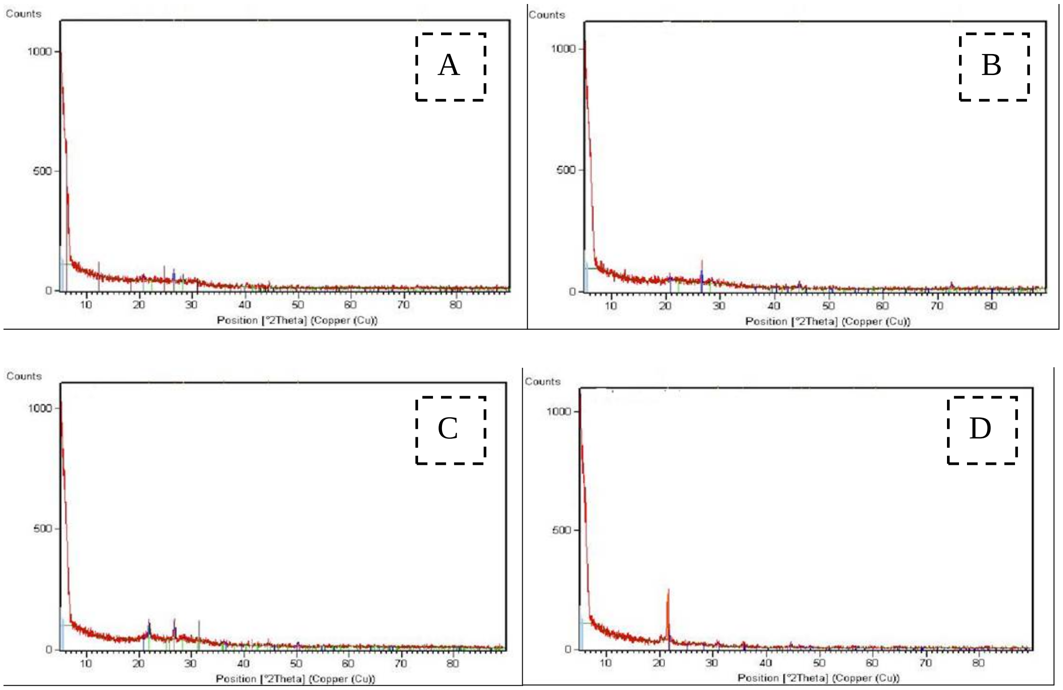

Fig. 5. The X-ray diffraction pattern of wheat hull ash obtained by burning at A) 500 °C B) 600 °C C) 700 °C D) 1000 °C for 5 h

Surface properties of wheat hull ash

SEM microstructure images of wheat hull ash samples burned at 400 °C**,** 500 °C, 600 °C, 700 °C, 800 °C, and 1000 °C for 5 h were obtained. Figure 6 provides SEM microstructure images obtained by using magnification ratio of 1000.

As can be seen in Figs. 6-A and 6-B, heterogeneous, diffused, thread-like structures are observed at low temperatures (400-500 °C). At 600-800 °C the ashes have a more porous and indented structure, which is eliminated at 1000 °C, and depression is observed on the structural skeleton, and it has a glass-like appearance. The adsorption capacity of the ash decreases as a result of the changes that occur in the structure of the ash obtained by burning at 1000 °C (Proctor 1990).

BET surface areas of wheat hull ash at different temperatures were measured and presented in Table 4. The study of the BET results of the ashes obtained by burning at various temperatures demonstrates that the surface area of the samples was increased, though slightly, up to 600 °C. This was explained by the fact that the volatile components in the biomass sample of wheat hull move away from the surface until the temperature reaches a certain value, thus causing a more porous and rough surface. The decrease in the surface areas of the ashes obtained by burning at temperatures above 600 °C can be explained by the completion of moving volatile components away from the sample. Additionally, the collapse occurring on the structural skeleton of the ash established porous, rough surface which was the result of almost complete consumption of carbon at higher temperatures, as can be seen from the SEM images 6-E and 6-F. Also, the decrease of surface area occurs due to the glass-like structure formation at higher temperatures.

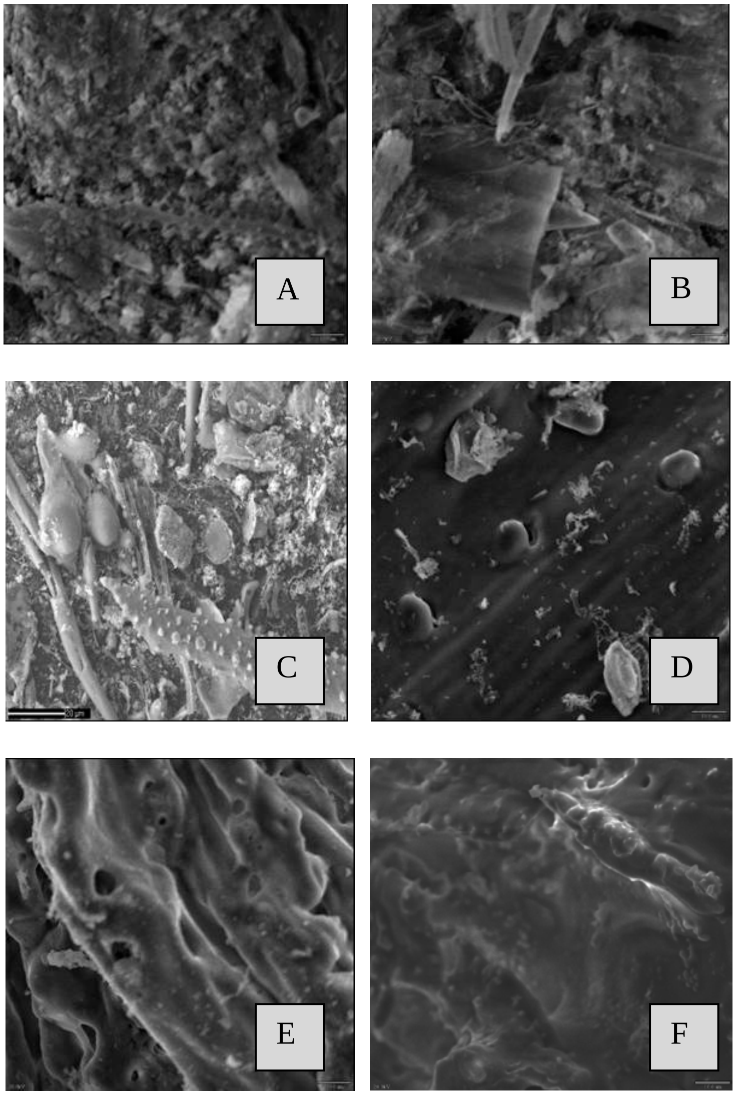

Fig. 6. SEM images of wheat hull ash obtained by burning at A) 400 °C B) 500 °C C) 600 °C D) 700 °C E) 800 °C F) 1000 °C for 5 h [1000x zoom]

Table 4. BET Surface Areas of Wheat Hull Ash Samples Obtained at Different Temperature Conditions

Temperature (°C)	BET Surface Area (m^2/g)
400	6.69
500	7.17
600	7.21
700	3.85
800	1.47
900	0.36
1000	0.11

Silica Xerogel Characterization

In Fig. 7 the major chemical groups present in silica are specified by the FTIR spectra. The broad band centered at 1062 cm^{-1} was attributed to the asymmetric stretching frequency of Si–O–Si, and the band centered at 795 cm^{-1} was due to the symmetric stretching of Si–O–Si. The small peak at 1650 cm^{-1} is attributed to the H – OH bending vibrations of the adsorbed water molecules.

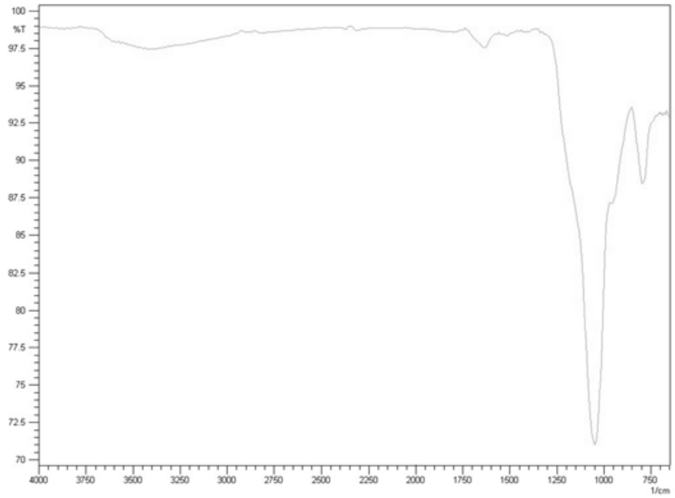

Fig. 7. FTIR spectrum of silica xerogel obtained from wheat hull ash

The XRF elemental analysis results of the produced silica xerogel showed that the major element was silica and the impurity was sodium chloride. The XRF results are means of triplicate analyses and they are presented in Table 5. The silica content of the silica xerogel was 93.9%. The Na_2O content of silica xerogel was 5.9%. The X-Ray diffraction pattern of silica xerogel obtained from wheat hull ash showed it to be amorphous, which was approved by the absence of any well-defined sharp peaks (Figure 8). Also, the peak at $2\theta = 32°$ and 46° corresponded to NaCl. Thus, the washing step will be made by extra deionized water in the further studies.

Table 5. Metal Oxide Content of Silica Xerogel Produced from Wheat Hull Ash

SiO_2 (%)	93.9
MgO (%)	0.09
Fe_2O_3 (%)	0.088
Na_2O (%)	5.9
MnO_2 (%)	0.032
CuO (%)	0.017

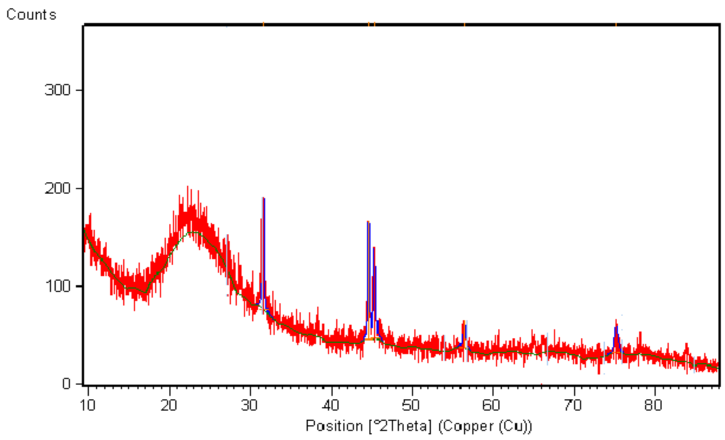

Fig. 8. XRD pattern of silica xerogel obtained from wheat hull ash

Figure 9 shows SEM images of silica xerogel. The silica xerogel particles were irregular in shape with a size that was generally in micron order (Figure 9 -D).

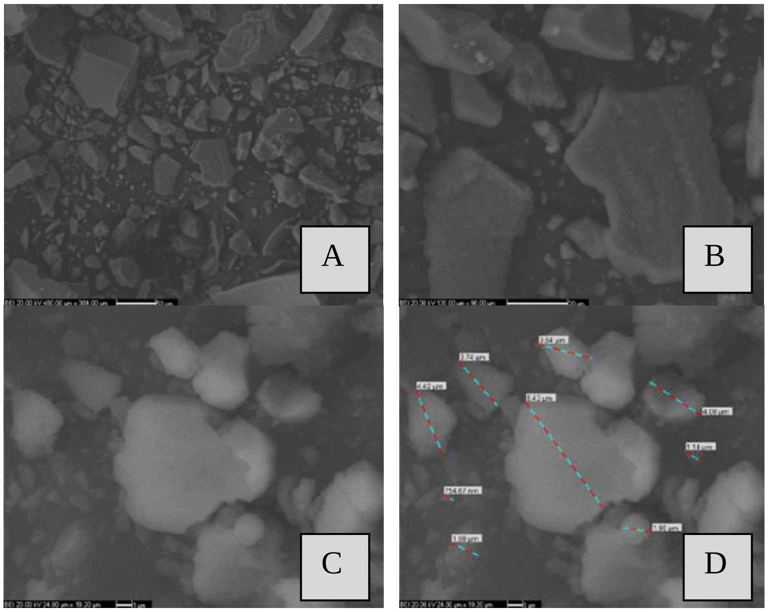

Fig. 9. SEM images of silica xerogel obtained from wheat hull ash A) 250x B) 1000x C) 5000x D) 5000x zoom

The yield of silica xerogel production has been calculated as 5.5 g silica xerogel/100 g of wheat hull.

CONCLUSIONS

This study showed the feasibility of utilizing wheat hull and its ash as a silica source since its characteristics are very useful to produce silica xerogel. The results of this study will make significant scientific contributions to the literature since there are not any detailed studies on wheat hull ash in literature. In conclusion, the present study has demonstrated that the ash obtained from wheat hull has a high content of silicon, and its characterization properties show that silicon can be produced from this source, which will create a brand new area of use for wheat hull and its ash. This work can be enhanced by investigating the effect of different parameters such as a washing agent and gelation pH on the silicate xerogel production. The results can be summarized as follows:

1. During the thermal treatment of wheat hull at different temperatures, the structure of ash's silica changes by temperature. Amorphous silica can be produced by choosing 600-700 °C as the ashing temperature.

2. The results show that the ashing temperature is an important factor to obtain an ash with a high SiO_2 content. Although the highest SiO_2 content was obtained when the ashing temperature set as 1000 °C, this temperature cannot be chosen as the optimum ashing temperature because the structure of silica (cristobalite) is not in a recoverable form.

3. Silica xerogel can be produced from wheat hull ash with 93.9% silica content. The yield of silica xerogel extraction from wheat hull was calculated as 5.5 g silica xerogel/100 g of wheat hull.

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