

SYNTHESIS OF MAGNESIUM SILICATE FROM WHEAT HUSK ASH: EFFECTS OF PARAMETERS ON STRUCTURAL AND SURFACE PROPERTIES

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In the present study, magnesium silicate was produced by using wheat husk ash. Wheat husk was burned at 600 °C to obtain an amorphous ash structure, and the ash was processed with sodium hydroxide solution with heat to extract silica. Sodium silicate solution and magnesium salts were used to synthesize magnesium silicate. The present study investigates effects of the feeding rate on magnesium silicate production (0.6 mL/min, 35 mL/min, 70 mL/min), the type of magnesium salt ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ or $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), temperature (25 °C or 50 °C), and the washing agent (water and acetone) on the chemical composition and surface characteristics of magnesium silicate. The results demonstrated that all of the variables affected the surface characteristics of magnesium silicate, such as surface area, particle size, and pore volume. However, it was also observed that the studied parameters did not affect the chemical composition of magnesium silicate. The wheat husk ash-based magnesium silicates obtained in the experimental study had a BET surface area ranging from 79 to 91 m^2/g and a particle size varying from 42 to 63 μm .

Keywords: Adsorbent; Magnesium silicate; Wheat husk; Wheat husk ash

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INTRODUCTION

Wheat is one of the most important grains with the highest sustenance value in the world. It has a remarkable consumption ratio due to its ease of cultivation, its ability to be converted into a variety of foods, its multipurpose use, and its role in the human diet. Every year, approximately 600 million tons of wheat is produced around the world (Boyacıoğlu 2009). A typical wheat kernel consists of a husk (pericarp) and seed. The total volume of husk stands for approximately 5% of the kernel, which consists of 6% protein, 2% ash, 20% cellulose, and 0.5% oil and non-starch polysaccharides (Hoseney 1994). The wheat husk contains in its structure carbon, oxygen, silica, potassium, and low levels of sulfur, phosphorus, sodium, magnesium, and aluminum (Bledzki *et al.* 2010).

Every year, large amounts of agricultural waste, known as “wheat husk”, are produced by the dehusking process of wheat (Motojima *et al.* 1995). Wheat husk, which is also produced during production of wheat and has a low economic value, can be used as a biomass source as a fuel in boilers because it has a calorific value of about 3.5 kcal/g (Bledzki *et al.* 2010; Zhang *et al.* 2009). As a byproduct of grains, husks are often burned in open air to produce energy, and the ashes are transferred to landfills, which cause significant environmental problems. However, silica (silicon) is available in the soil in large amounts, and it accumulates in the tissues of root plants, and therefore, some husk and husk ash, such as rice husk, which contains silica, can be

recovered in the industry. For instance, there have been numerous studies on the use of rice husk for production of silica-based compounds that contain ceramic, silicon carbide, silicon nitride, silicon tetrachloride, Si_3N_4 whiskers pure silicon, silica gel, sodium silicate, and zeolite (Motojima *et al.* 1995; Kalapathy *et al.* 2000; Sun and Gong 2001). However, there have been very few studies in the literature that focus on the recovery of wheat husk and ash as a source of silicon. A study was carried out to produce silicon with a porous nanostructure from wheat stalk (Chen *et al.* 2010). Wheat husk is often used in the construction industry as a concrete additive (Zhang and Khatib 2008). Goyal *et al.* (2007) used rice husk ash and wheat straw ash to produce ash-substituted mortar. The study shows that wheat straw ash (74.23 %) and rice husk ash (90.11 %) have high silica content. According to this result, wheat husk ash can be used for production of silica-based products as rice husk ash. Recovery of wheat husk may bring economical benefits in the future as well as contributing to efforts of waste management by reducing environmental pollution.

Synthetic magnesium silicate is an important adsorbent. It has a vast area of use as an industrial or analytical adsorbent. It is generally used to purify biodiesel because it has a high capacity to adsorb polar (free acid, partial glyceride, *etc.*) components (Yates and Caldwell 1993; Yates *et al.* 1997; Buczek and Chwialkowski 2004; Cooke *et al.* 2005; Taspinar and Ozgul-Yucel 2008). Sodium silicate solution and magnesium salts are used as raw materials to produce synthetic magnesium silicate. Production of sodium silicate conventionally requires high energy because it is obtained from the reaction of quartz sand with sodium carbonate at 1300 °C (Kalapathy *et al.* 2002). As an alternative to this process, Kamath and Proctor (1998) found a new, lower-energy method for the production of sodium silicate from rice husk ash. Their study demonstrated that the silica that was available in rice husk ash was amorphous at 600 °C and lower temperatures, and it could be easily extracted under alkali conditions. Therefore, rice husk ash, which includes silica, was boiled with sodium hydroxide in order to produce sodium silicate.

Ozgul-Yucel *et al.* (2004) also used the method of Kalapathy and Proctor, and obtained sodium silicate solution from rice husk ash that contained 77% silica. A certain ratio of magnesium sulfate salt was added to the obtained sodium silica solution at room temperature, which yielded magnesium silicate with a surface of 245 m²/g in the form of white precipitate.

Varol (2006) obtained magnesium silicate from rice husk ash but used a higher amount of water for dilution in reaction medium. The researcher reports that adding more water to the reaction medium at room temperature prevented the co-precipitation of sodium sulfate and sodium chloride salts from the magnesium sulfate or magnesium chloride treatment. The magnesium silicate obtained with this method had a higher surface area than the commercial magnesium silicate called Magnesol.

The present study aimed to produce magnesium silicate from wheat husk ash for the first time. The experimental variables, which affected the reaction, were temperature, feeding rate of magnesium salt, type of magnesium salt ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), and washing solvent (water and acetone). The effect of these parameters on properties of magnesium silicate, such as surface area, chemical composition, and particle size, were investigated.

MATERIALS AND METHODS

Materials

Wheat husk was supplied by Doruk Marmara Flour Factory located in the Marmara region of Turkey. Wheat husk had moisture and ash contents of 7.7% and

9.95%, respectively, which were determined according to the methods of Nielsen (1993). The chemicals used in production of magnesium silicate, which were analytical reagent grade, NaOH, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, HCl, and acetone, were supplied by Merck (Darmstadt, Germany). Besides, Florisil from Sigma was used for comparison with magnesium silicate. An ash furnace (Protherm, Turkey) was used for the burning process.

Preparation of Sodium Silicate Solution

The silicate solution was prepared according to the method of Kalapathy and Proctor (2000). The wheat husk obtained from a flour factory was burned in open air with a hand-lighter. Semi-burned wheat husk was burned again in an incineration furnace (Protherm, Turkey) at 600 °C for 5 hours because it is known that ashes with high silica content are obtained at temperatures lower than 400 °C (Kamath and Proctor 1998). Ozgul-Yucel *et al.* (2004) also demonstrated that rice husk ash has an amorphous structure when burned at 600 °C. Thus, 600 °C was chosen as the burning temperature. 100 grams of ash were placed into a 2 L flask, 600 milliliters of 1 M sodium hydroxide solution was added, and the flask was capped with a watch glass. The mixture was boiled for one hour with constant stirring using a magnetic stirring bar. Then the mixture was filtered under vacuum, and the upper carbon residues were washed with 50 mL of hot water to separate clear sodium silicate solution from carbon residues (Ozgul-Yucel *et al.* 2004). The silicate solution was preserved in a plastic bottle until used.

Determination of silica content of the sodium silicate solution was achieved by lowering the pH of the solution from 12 to 7 to precipitate silica. After filtration, silica solution was double washed with 100 mL of water, dried at 110 °C for 12 h, and weighed (Kamath and Proctor 1998).

Preparation of Magnesium Silicate

The synthesis procedure of magnesium silicate powder is given in Fig. 1. Production of magnesium silicate consists of four steps, which are reaction, filtration, washing for eliminating impurities, and drying at room temperature. Production of magnesium silicate was performed at 25 °C or 50 °C using two types of magnesium salts ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ or $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$). A stoichiometric quantity of magnesium salt was completely dissolved in distilled water and was poured into a double-jacketed 350 mL reactor. Then the sodium silicate solution (Na_2SiO_3) was added via a peristaltic pump (Heildolph Pumpdrive 5201, Germany) at a feeding rate of 0.6 mL/min, 35 mL/min, or 70 mL/min. During the reaction, a mechanical stirrer constantly stirred the solution. After the reaction was completed, the mixture was filtered under vacuum. The residual magnesium silicate on the filter paper was washed with 500 mL of distilled water to remove impurities. In only one experimental set was the upper part was washed with 200 mL of acetone after washing with 500 mL of water to see the effects of polar solution on the production of magnesium silicate to eliminate organic impurities. The solid part, which was obtained by filtration, was dried at room conditions and came to balance.

For production from wheat husk ash, 25 mL sodium silicate solution (4.17 g wheat husk ash, containing 2.44% SiO_2), 4.242 grams of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ or 5.166 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, and 75 mL dissolving water were used in each batch. The amount was calculated stoichiometrically according to the reaction Equation 1:



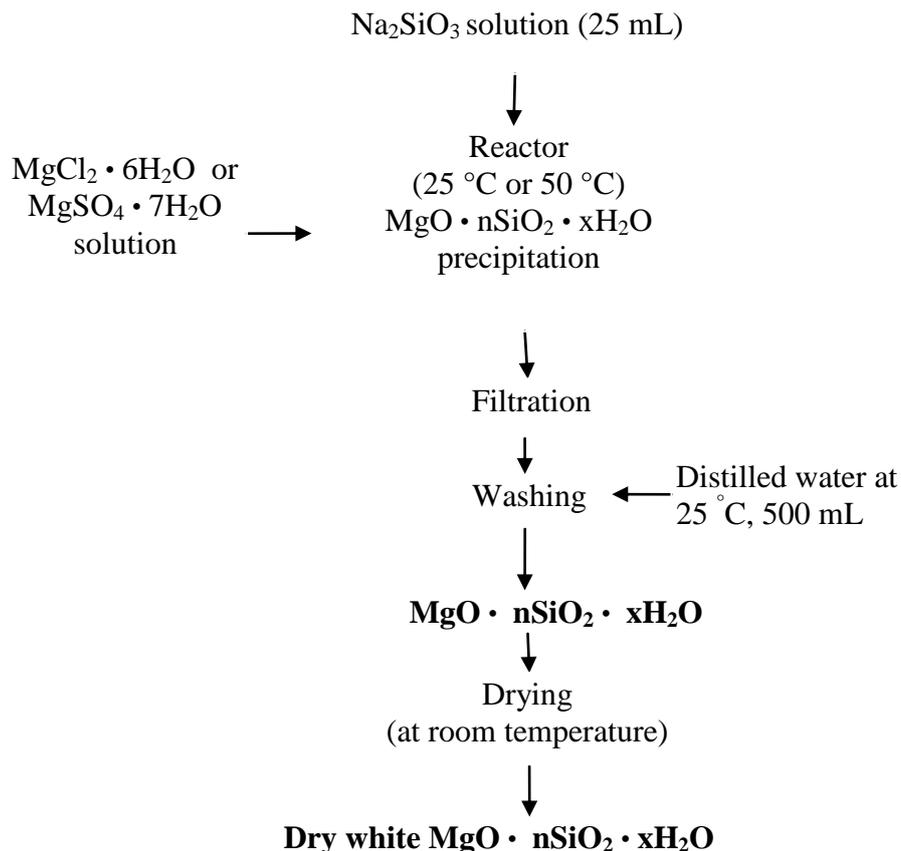


Fig. 1. Production of $\text{MgO} \cdot n\text{SiO}_2 \cdot x\text{H}_2\text{O}$ powder in each batch

Characterization of Wheat Husk and Wheat Husk Ash

Fourier transform infrared spectra of wheat hull were obtained with the use of a SHIMADZU, IR Prestige 21; USA in the range of 600 to 2000 cm^{-1} .

The chemical properties of wheat husk ash were determined by using an Inductively Coupled Plasma (ICP) system (Perkin Elmer Optical Emission Spectrometer Optima 2100 DV, USA). X-ray diffraction patterns of wheat husk ash were obtained with an X'Pert PRO PANalytical (Holland) XRD system using an acceleration voltage of 40 kV and current of 45 mA . The diffraction angle was scanned from 5° to $90^\circ 2\theta$, with a step size 0.003 .

Characterization of Magnesium Silicates

Determination of surface properties and micropore volumes of the obtained magnesium silicates and commercial magnesium silicate (Florisil) was achieved by the use of SEM images (JEOL JSM 5410 LV, Japan) and BET measurements (Costech 1042 Sorptometer, Italy). The Fourier transform infrared spectra were measured with a SHIMADZU, IR Prestige 21 (USA) device in the range of 600 to 2000 cm^{-1} .

The particle size of magnesium silicate was determined by a Laser diffraction particle size analyzer (Malvern 2000, England). The SiO_2 content of the magnesium silicate produced was determined by the wet analysis method and atomic absorption spectrophotometer (Furman 1968) (PerkinElmer 110 B, USA). The humidity content of magnesium silicates was determined according to Yates and Caldwell (1993). The dry base chemical composition and loss on ignition of magnesium silicates were determined according to FAO JECFA Monographs 11.

RESULTS AND DISCUSSION

Properties of Husk

The ash content of wheat husk was found to be 9.95 %. Thus, the recovery of wheat ash was 99.5 kg ash / kg of wheat husk.

Figure 2 shows the FTIR spectrum of wheat husk. The spectrum exhibits the peak of the O-Si-O band at 1032 cm^{-1} . The peak at 1631 cm^{-1} was due to the H – OH bending vibrations of the adsorbed water molecules. An Si – OH stretching vibration was observed at 920 cm^{-1} . On the other hand, the peak at about 1726 cm^{-1} is due to the C=O stretching of the acetyl and uronic 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 peaks at around 1640 cm^{-1} were related to the amide I band that represents 80% of the C=O stretching of the amide group, coupled to the in-plane N–H bending and C–N stretching modes. The region of 1450 to 1370 cm^{-1} represents the C–H symmetric and asymmetric deformations. The small peak at around 900 cm^{-1} is characteristic of β glycosidic linkages between the sugar units (Bledzki *et al.* 2010).

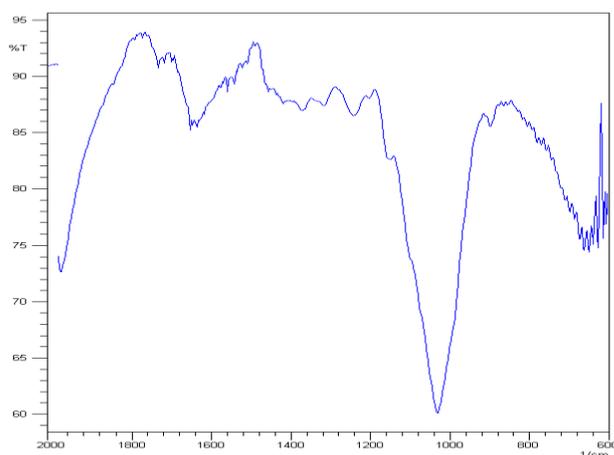


Fig. 2. FTIR spectrum of wheat husk

Properties of Ash

The chemical content of ash is given in Table 1.

Table 1. The Chemical Composition of Wheat Husk Ash Obtained at $600\text{ }^{\circ}\text{C}$

Compound	Value (%)
SiO ₂	43.22
K ₂ O	11.30
MgO	0.99
Fe ₂ O ₃	0.84
Na ₂ O	0.16
Cr ₂ O ₃	0.0004
MnO ₂	0.02
CaO	5.46

The ash was composed of SiO_2 , K_2O , MgO , Fe_2O_3 , Na_2O , Cr_2O_3 , MnO_2 , and CaO . The major component of the ash was SiO_2 with 43.22%. Other major components were found as K_2O (11.3%) and CaO (5.46%). The X-Ray diffraction pattern of wheat husk ash obtained at 600 °C is given in Fig. 2, which demonstrates that the ash had an almost completely amorphous structure. The SiO_2 content of sodium silicate solution, which was obtained by NaOH extraction of the ash burned at 600 °C to be used in the experiments, was found to be 2.44%.

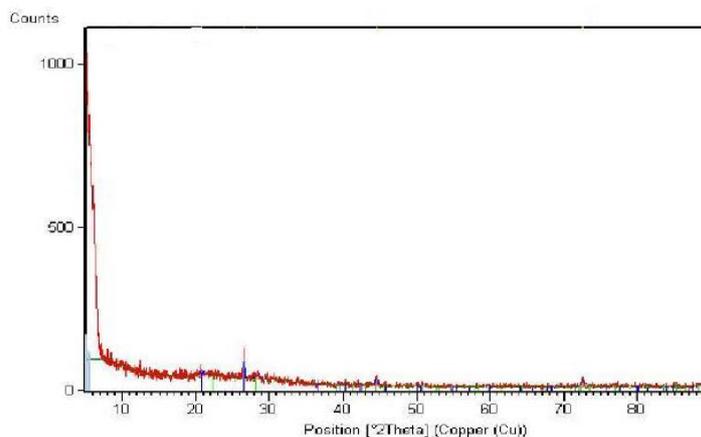


Fig. 3. X-Ray diffraction pattern of wheat husk ash obtained at 600 °C

Effect of Sodium Silicate Solution Feeding Rate on Magnesium Silicate Production

In reaction crystallization, a solution of one reactant is mixed with a solution of the other, and the crystallizing substance is formed by a chemical reaction in concentrations exceeding the solubility. Frequently the reaction is fast or very fast, and the mixing conditions influence the product size distribution significantly (Aslund and Rasmuson 1992). Feeding rate, which is the total feed time, might have a significant role in surface properties of particles such as shape and particle size distribution. To observe these effects in production of magnesium silicate, the process was performed at 25 °C with three different magnesium salt feeding rates, which were 0.6 mL/min, 35 mL/min, or 70 mL/min. $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ was used for this production batch. The results are given in Table 2. The feeding rate at 35 mL/min gave a surface area of 87.41 m^2/g , and the feeding rate of 70 mL/min yielded a surface area of 79.13 m^2/g . The maximum BET surface area (91.74 m^2/g) was achieved at the feeding rate of 0.6 mL/min. However, working with low feeding rates will not be practical and efficient, as it may be time-consuming. It is more convenient to work at higher feeding rates, since there is not a significant difference between low and high feeding rates in terms of surface area. Decreasing the feeding rate from 70 mL/min to 0.6 mL/min slightly increased the surface area.

The magnesium silicates have an aggregate structure, which is demonstrated in all SEM images presented in Fig. 4. Since magnesium silicates have an aggregate structure, the particle size of the produced magnesium silicates can also be defined as the aggregate particle size. There were not any significant differences between SEM images of 0.6 mL/min feeding rate and SEM images of 70 mL/min feeding rate. The chemical composition of magnesium silicates produced was examined. In Table 2, it can be clearly seen that the feeding rate did not affect the chemical composition of the magnesium silicates.

The BET surface area of commercial magnesium silicate, Florisil, was found as 206.96 m^2/g . Therefore, the BET surface area of magnesium silicates obtained by using

wheat husk ash were smaller than the values for commercial magnesium silicate. Taspinar and Ozgul-Yucel prepared magnesium silicate from rice husk ash and found a high surface area ($245 \text{ m}^2/\text{g}$) at 25°C (Taspinar and Ozgul-Yucel 2008).

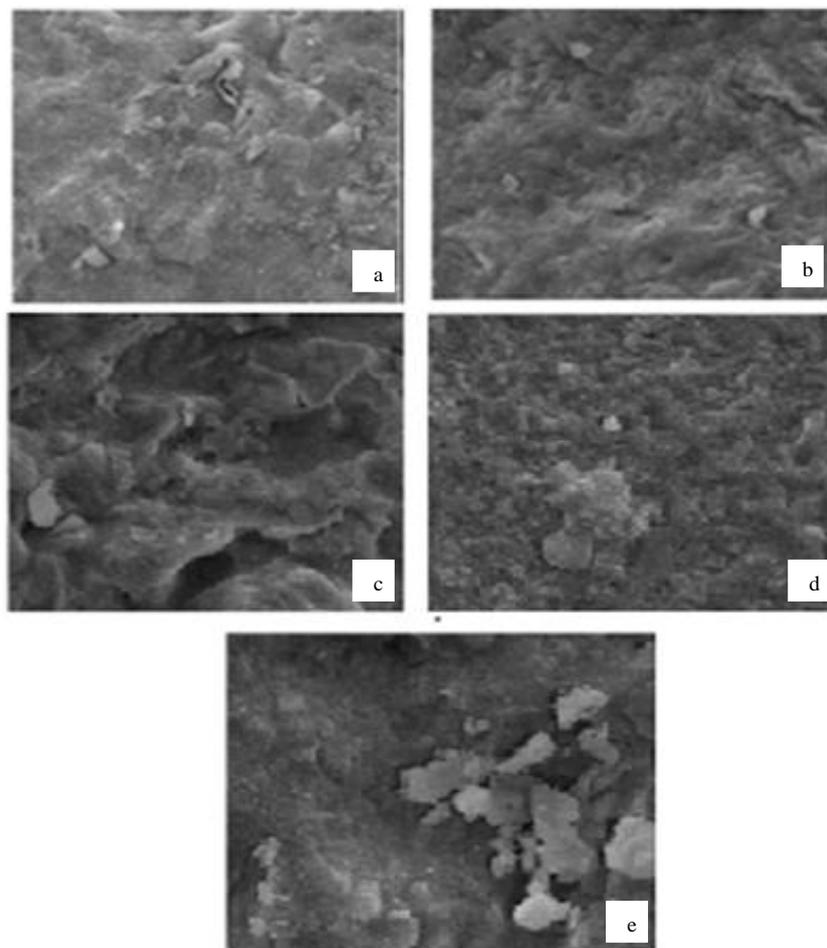


Fig. 4. SEM images of magnesium silicates (1000x)

- a) Obtained with 0.6 mL/min feeding rate at 25°C by $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt.
- b) Obtained with 70 mL/min feeding rate at 25°C by $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt.
- c) Obtained with 35 mL/min feeding rate at 50°C by $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt.
- d) Obtained with 35 mL/min feeding rate at 25°C by $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ salt.
- e) Obtained with 35 mL/min feeding rate at 25°C by $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and washed with acetone.

Table 2. Effect of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ Feeding Rate on Magnesium Silicate Production

Feeding Rate (mL/min)	BET Surface Area (m^2/g)	Chemical Content (%)					
		SiO_2	MgO	CaO	Fe_2O_3	Na_2O	K_2O
0.6	91.74	42.6	7.82	n/a	0.03	0.05	0.13
35	87.41	42.5	7.92	n/a	0.07	0.07	0.21
70	79.13	42.4	7.99	n/a	0.05	0.05	0.15

Furthermore, FTIR the spectrum of magnesium silicate obtained with a 0.6 mL/min feeding rate at 25 °C by using $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt and the FTIR spectrum of Florisil are provided in Fig. 5. Similar peaks can be observed on both spectra, and the strong peak at 1000 cm^{-1} and peaks near 980 cm^{-1} and 620 cm^{-1} are the Si-O stretching vibrations. The very weak absorptions near 1240 cm^{-1} are ascribed to Si-O vibrations. Bands near 750 cm^{-1} are assigned to Si-O-Si bending vibrations. The vibrations of near 1610 cm^{-1} are OH stretching vibrations.

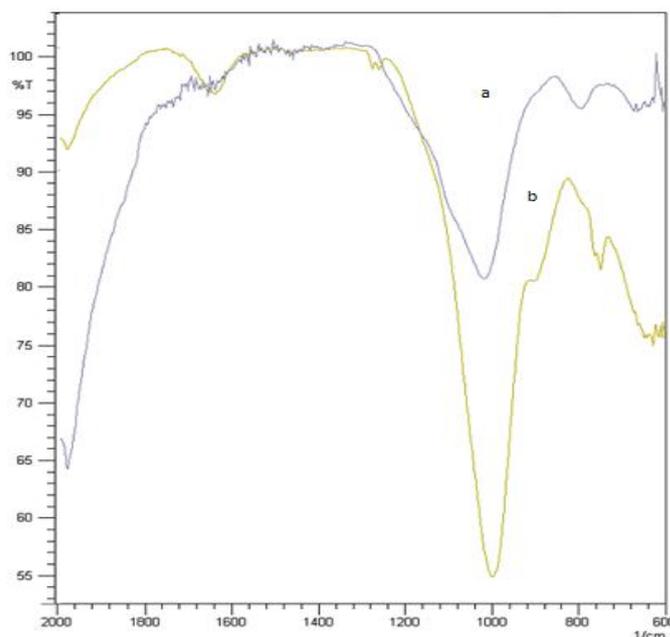


Fig. 5. FTIR spectra of a) commercial magnesium silicate (Florisil) and b) magnesium silicate obtained with 0.6 mL/min feeding rate at 25 °C by $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ salt

Effect of Magnesium Salt Type on Magnesium Silicate Production

$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solutions were individually used as the reactant at 35 mL/min feeding rate at 25 °C to identify the effects of magnesium salt. The properties of the magnesium silicates obtained are presented in Table 3. The BET surface area of magnesium silicate obtained with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ solution was greater than the one produced with $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution. The BET values of magnesium silicates obtained with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solutions were $87.41\text{ m}^2/\text{g}$ and $80.19\text{ m}^2/\text{g}$, respectively.

The SiO_2 content of the produced adsorbents was almost the same; however, the MgO content of magnesium silicate that was produced with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ solution was slightly higher. The particle size distribution of magnesium silicates obtained with $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ salt with 35 mL/min feeding rate at 25 °C is given in Fig. 3d. The average particle size of magnesium silicate obtained with $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution was 50 μm , which is higher than the one produced with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ solution (42 μm).

The SEM image of magnesium silicate obtained by using $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ salt with 35 mL/min feeding rate at 25 °C is given in Fig. 4d. In the literature, Taspınar and Ozgul-Yucel (2008) reported similar results for magnesium silicates that were obtained from rice husk ash. The BET surface area of magnesium silicate obtained by using $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution ($641\text{ m}^2/\text{g}$) was lower than the one obtained with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ solution ($680\text{ m}^2/\text{g}$).

Table 3. The Effect of Magnesium Salt, Temperature, and Washing Solvent on Magnesium Silicate Production

Magnesium Salt	Feeding Rate (ml/min)	BET Surface Area (m ² /g)	Average Particle Size (µm)	Micropore Volume (mm ³ /g)	Chemical Content (%)				
					SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O
MgCl ₂ ·6H ₂ O	35	87.41	42	11.01	42.5	7.92	0.07	0.07	0.21
MgSO ₄ ·7H ₂ O	35	80.19	50	5.38	42.4	6.78	0.02	0.06	0.14

The dry base chemical composition of magnesium silicates is presented in Table 4. Precipitated magnesium silicates had a lower magnesium oxide and silica content than that of the commercial magnesium silicates. However, loss on ignition and moisture content of the samples comply with the limit values of commercial magnesium silicate.

Table 4. Dry Base Compositions, LOI, and Humidity Content of Obtained Magnesium Silicates with 35 mL/min Feeding Rate at 25 °C

	SiO ₂ (%)	MgO (%)	LOI ^a (%) (dry base)	Humidity Content (%)
Commercial Magnesium Silicate (Anonymous 2009)	67	15	15 (max)	20 (max)
MgCl₂ · 6H₂O	50	9.31	14.15	15
MgSO₄ · 7H₂O	49.88	7.97	14.02	14.9

^a Percentage additional weight loss on ignition at 900 °C for 20 min. (percentage of dry basis weight).

Effect of Reaction Temperature on Magnesium Silicate Production

The production was carried out at 25 °C and 50 °C by using MgCl₂ · 6H₂O solution with a 35 mL/min feeding rate to observe the effect of temperature on production of magnesium silicate. A comparison of production processes at different temperatures is provided in Table 5. It can be clearly seen from the table that the BET surface area of the production at 25 °C was greater than in the case of production at 50 °C. It was determined that the increase of synthesis temperature provides a better

reaction, and so, leads to an increase of average particle size and decrease of the BET surface area of magnesium silicate.

Table 5. The Effect of Reaction Temperature on Magnesium Silicate Production

Magnesium Salt	Reaction Temperature (°C)	BET Surface Area (m ² /g)	Average Particle Size (µm)	Micropore Volume (mm ³ /g)	Chemical Content (%)				
					SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O
MgCl ₂ ·6H ₂ O	25	87.41	42	11.01	42.5	7.92	0.07	0.07	0.21
MgCl ₂ ·6H ₂ O	50	82.00	48	2.34	41.5	7.77	0.02	0.03	0.18

The particle size distribution diagram is given in Fig. 6. The average particle size of magnesium silicate produced at 25 °C was found to be slightly smaller. The particle size distribution of magnesium silicates produced at 50 °C is presented in Fig. 6b. The SEM image of the sample is shown in Fig. 4c. In terms of chemical composition, the SiO₂ and MgO contents of magnesium silicate produced at 25 °C were quite similar to the magnesium silicate obtained at 50 °C.

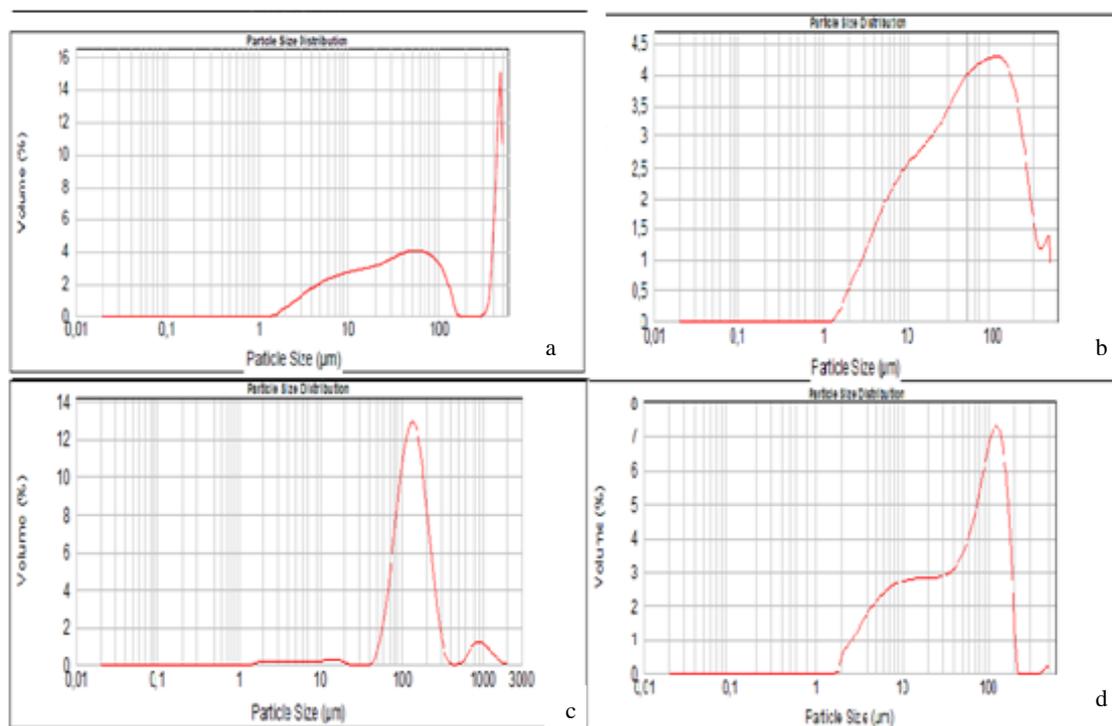


Fig. 6. Particle size distributions of magnesium silicates
 a) Obtained with 35 mL/min feeding rate at 25 by MgCl₂ · 6H₂O salt.
 b) Obtained with 35 mL/min feeding rate at 50 by MgCl₂ · 6H₂O salt.
 c) Commercial Florisil
 d) Obtained with 35 mL/min feeding rate at 25 °C by MgSO₄ · 7H₂O salt.

The yield of magnesium silicate was similar in each production and has been calculated as 15 g magnesium silicate / 100 g of wheat husk.

Effect of Washing with Polar Solvent on Production of Magnesium Silicate

Magnesium silicate was obtained with a 35 mL/min feeding rate at 25 °C and by using $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. In all trials until this step, the magnesium silicate obtained was washed with distilled water to remove impurities. After washing with water, the magnesium silicate obtained was washed with 200 mL acetone to observe the effect of washing with acetone on magnesium silicate production.

The properties of magnesium silicates obtained after washing with water or acetone are given in Table 6. Since acetone is a volatile organic compound, when it came into contact with magnesium silicate, the water in the pores was removed. Washing with acetone caused a significant decrease in the BET surface area of magnesium silicate ($39.2 \text{ m}^2/\text{g}$) because of higher agglomeration. The particle size distribution of magnesium silicates could not be identified because magnesium silicates became lumpy after washing with acetone. However, acetone did not affect crystallization. Therefore, it was considered that magnesium silicates obtained by washing with acetone had the same particle size as those obtained by washing only with water under the same conditions. The SEM image of magnesium silicate that was obtained by washing with acetone is given in Fig. 4e. Agglomeration that was caused by the increased effect of acetone on drying process can be clearly seen in Fig. 4e. Washing with acetone did not make any difference in the chemical composition of magnesium silicate (Table 6). It was concluded that washing with acetone was not necessary for production of magnesium silicate since acetone made a negative effect on the surface area.

Table 6. Effect of Washing with Polar Solvent on Magnesium Silicate Production

Magnesium Salt	Washing Solvent	BET Surface Area (m^2/g)	Average Particle Size (μm)	Micropore Volume (mm^3/g)	Chemical Content (%)				
					SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	Water	87.41	42	11.01	42.5	7.92	0.07	0.07	0.21
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	Acetone	39.20	-	9.39	42.5	7.56	0.02	0.09	0.25

CONCLUSIONS

Various reaction conditions were studied in an effort to easily and rapidly obtain a well-adsorbing magnesium silicate from wheat husk ash by alkali extraction. It was found that the feeding rate, temperature, magnesium salt type, and washing agent affected the surface area and agglomerate particle size of magnesium silicates. It was also observed that use of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ as a magnesium salt gave a recovery of magnesium silicate with a greater surface area than the use of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. The chemical compositions of precipitated magnesium silicates were not affected by any factors investigated. The factor to be focused for magnesium silicate with greater BET

surface area and chemical composition is the reagent mole ratio. In addition, the effect of different mobilizing agents such as n-propyl amine, polyethylene glycol can be tried for further investigation. Wheat husk is a potential green source and one of the main agricultural crops of Turkey. With this method, recovery of wheat husk can help to settle the problems in waste disposal as well as contribute to recovery of industrial products. Also, the results on the effect of experimental parameters such as type of reactant, temperature, flow rate, and washing will be very useful in industrial standpoint. On the other hand, mixture of wheat husk ash and rice husk ash, and other agricultural wastes containing silica can be evaluated for magnesium silicate production in the future studies.

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REFERENCES CITED

- Anonymous. (2009). "Magnesol, synthetic magnesium silicate," January 10, 2009, Dallas Group of America, Inc.: <http://www.dallasgrp.com>
- Aslund, B. L., and Rasmuson, A. C. (1992). "Semibatch reaction crystallization of benzoic acid," *AIChE J.* 38, 328-342.
- Bledzki, A. K., Mamuna, A. A., and Volk, J. (2010). "Physical, chemical and surface properties of wheat husk, rye husk, and soft wood and their polypropylene composites," *Composites: Part A* 41, 480-488.
- Boyacıoğlu, H. (2009). *World Wheat Condition 2009*, Feed Management Systems.
- Buczek, B., and Chwialkowski, W. (2004). "Purification of used frying oil by treatment with magnesium silicate," 3. Euro Fed Lipid Congress, Edinburg. 5-8 September.
- Chen, H., Wang, F., Zhang, C., Shi, Y., Jin, G., and Yuan, S. (2010). "Preparation of nano-silica materials: The concept from wheat straw," *J. Non-Crystalline Solids* 356, 2781-2785.
- Cooke, B., Bertram, B., and Abrams, C. (2005). "Purification of biodiesel using synthetic magnesium silicate," 96th AOCS Annual Meeting and Expo, Utah, 1-4 May.
- FAO JECFA Monographs 11 (2011), *Compendium of Food Additive Specifications*, Food and Agriculture Organization of the United Nations Rome, 2011, ISSN 1817-7077.
- Furman, N. W. (1968). *Standard Methods of Chemical Analysis*, 6th Ed., D. Van Nostrand, Princeton, 1, 953.
- Goyal, A., Kunio, H., Ogata, H., Garg, M., Anwar, A.M., Ashraf M., and Mandula (2007). "Synergic effect of wheat straw ash and rice-husk ash on strength properties of mortar," *J. Appl. Sci.*, 7: 3256-3261.
- Hoseney, C. R. (1994). *Principles of Cereals-Sci. and Technol.*, 2nd Ed., AACC, St.Paul, Minnesota, USA.
- Kamath, S. R., and Proctor, A. (1998). "Silica gel from rice husk ash: Preparation and characterization," *Cereal Chem.* 75, 484-487.
- Kalapathy, U., Proctor, A., and Shultz, J. (2000a). "A simple method for production of pure silica from rice husk ash," *Bioresource Technol.* 73, 257-262.

- Kalapathy, U., Proctor, A., and Shultz, J. (2000b). "Production and properties of flexible sodium silicate films from rice husk ash silica," *Bioresource Technol.* 72, 99-106.
- Kalapathy, U., Proctor, A., and Shultz, J. (2002). "An improved method for production of silica from rice husk ash," *Bioresource Technol.* 85, 285-289.
- Motojima, S., Hori, Y., Gakei, S., and Iwanaga, H. (1995). "Preparation of Si₃N₄ whiskers by reaction of wheat husks with NH₃," *J. Mat. Sci.* 30, 3888-3892.
- Nielsen, S. S. (1993). *Food Analysis*, 2nd Ed., A Chapman & Hall Food Science Title, An Aspen Publication, Gaithersburg, Maryland.
- Ozgul-Yucel, S., Tolay, M., Erdag, S., and Turkyay, S. (2004). "Magnesium silicate synthesis from rice husk ash," *J. Am. Oil Chem. Soc.* 81, 619-620.
- Sun, L., and Gong, K. (2001). "Silicon-based materials from rice husks and their applications," *Ind. Eng. Chem. Res.* 40, 5861-5877.
- Sun, X. F., Xu, F., Sun, R. C., Fowler P., and Baird, M. S. (2005). "Characteristics of degraded cellulose obtained from steam-exploded wheat straw," *Carbohydrate Res.* 340, 97-106.
- Taspinar, O. O., and Ozgul-Yucel, S. (2008). "Lipid adsorption capacities of magnesium silicate and activated carbon prepared from the same rice husk," *Eur. J. Lipid Sci. Technol.* 110, 742-746.
- Varol, D. (2006). "Production of magnesium silicate from rice husk ash," M.Sc. Thesis, Istanbul Technical University, Istanbul, Turkey.
- Yates, R. A., and Caldwell, J. D. (1993). "Regeneration of oils used for deep frying: A comparison of active filter aids," *J. Am. Oil Chem. Soc.* 70(5), 507-511.
- Yates, R. A., Caldwell, J. D., and Perkins, E. G. (1997). "Diffuse reflectance Fourier transform infrared spectroscopy of triacylglycerol and oleic acid adsorption on synthetic magnesium silicate," *J. Am. Oil Chem. Soc.* 74, 289-292.
- Zhang, J. S., and Khatib, J. M. (2008). "Using wheat husk ash waste in the production of a sustainable real estate construction material," *Real Estate Develop. Econ. Res. J.* 1(1), 18-29.
- Zhang, J. S., Khatib, J. M., Booth, C., and Siddique, R. (2009). "Possible utilization of wheat husk ash waste in the production of precast concrete elements," Excellence in Concrete Construction through Innovation, Proceedings of the conference held at the Kingston University, United Kingdom, 9 - 10 September 2008, ISBN: 978-0-415-47592-1.

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