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# Experimental Investigation of Temperature and Reaction Time for Preparation of Silica from Wheat Husk

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## ABSTRACT

*Wheat is one of the major crops grown throughout the world. Once the paddy is separated from Wheat grain, the outer kernel (hull) is removed from rest of the grain. This constitutes about, one-third of the total mass of grains, commonly termed as “Wheat husk” or “Wheat hull”. The annual Wheat husk produced in India amounts generally approximately 713 million tonnes. Wheat husk is generally not recommended as cattle feed since its cellulose and other sugar contents are high. Wheat husk has a high ash content varying from 20-22% and Silica is the major constituent of Wheat husk ash varying from 60-65%. With such large silica content in the Wheat husk it becomes economical to extract silica from the ash, which has wide market and also takes care of ash disposal.*

*For the study presented here an attempt was made to investigate a simple process to manufacture precipitated silica from this waste Wheat husk. Kinetic study and evaluation of rate constant (both theoretical and experimental) from kinetic data obtained from experiments had been carried out to find the optimum temperature and reaction time for the reaction. Experimental data shows better performance as well as easy industrial implementation of the process.*

### Keywords

*Wheat husk, Wheat husk ash, sodium silicate, Silica, rate constant.*

## 1. INTRODUCTION

Wheat husk is an agricultural waste material and are often left in the field, burnt in the open or a small portion is used as a component in animal beddings. The fact is a cheap and abundant source of silica remains largely unrealized. To some extent, wheat husk has been utilized as fuel for cooking in some developing country, but it is neither fully nor efficiently utilized. Such underutilization clearly shows the wastage and loss of resources which in reality can generate revenue through the recovery of silica via methods such as combustion [1].

Wheat husk ash is one of the silica rich raw materials containing about 45-50% silica (after complete combustion) among the family of other agro wastes. From a rough estimation the production of paddy in the country about seven hundred thirteen million tons of wheat husk is obtained from wheat mills. Using wheat husk as a raw material five million tons of pure high grade silica can be produced to meet the high demand of various industries that are using it. The main states with large number of wheat mills are Uttar Pradesh, Punjab, Madhya Pradesh, Haryana, Maharashtra, etc. [1, 2].

The chemical process discussed in this work not only provides a solution for waste disposal but also recovers a valuable silica product, together with certain useful associate recoveries. Silica ( $\text{SiO}_2$ ) is one of the valuable inorganic multipurpose chemical compounds. It can exist in gel, crystalline and amorphous forms. However, manufacture of pure silica is energy intensive. A variety of industrial processes, involving conventional raw material (like sand) required high furnace temperature (more than  $500^\circ\text{C}$ ) [3]. Recently a large amount of precipitated silica has been extracted from various types of bio waste such as wheat husk ash, Rice husk ash and bagasse ash etc. Common sources of silica are Wheat husks, Rice husk, Sand, Bamboo leaves. The study presented here used a non-conventional raw material method of manufacturing silica by

reacting wheat husk ash with caustic soda. The solution of silica so formed is then filtered from unreacted, unburned carbon particles, originally present in the ash and finally concentrated in evaporator [4].

## 2. THEORY

**2.1 Conventional process:** Silica is obtained from quartz sand. The conventional process of manufacturing sodium silicate is through the fusion of silica sand with high purity soda ash in furnaces at high temperatures (1300°C –1500°C) forming water glass (a solid) which is then crushed and dissolved in water and digested under pressure with steam [5,6]. Therefore the potential for obtaining this expensive sodium silicate solution through the relatively less costly process of caustic digestion of wheat husk ash had been reported by Stephens et al. (2003).the production of water glass through this route formed the foundation of all commercial processes for making sodium or other soluble silicate solutions today.

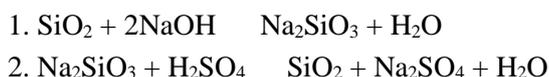


Therefore the potential for obtaining this expensive sodium silicate solution through the relatively less costly process of caustic digestion of wheat husk ash had been reported by Stephens et al. (2003)[7].

**2.2 Process used for experimental work:** In this process, silica is manufactured using wheat husk ash as a source of silicate or silica and ash is then reacted with sodium hydroxide which yields the sodium silicate, which on further reacting with sulphuric acid gives silica along with by product sodium sulphate. The basis for this proposed process is the wheat husk ash with higher silica content [8]. The process to extract all the silicate contents from the wheat husk is just by burning the wheat husk under controlled conditions of air and temperature. By this condition we can get the clear white ash in the furnace. The basic steps in the production of precipitated Silica are:

1. Obtaining WHA from wheat husk.
2. Dissolution of WHA in alkali.
3. Precipitation of silica from silicate solution.

The reactions involved are;



From the above comparison we see that all operations are carried out at temperature less than 100°C. Treating Wheat Husk Ash with acids is an exothermic reaction. The energy from this exothermic reaction and combustion of Wheat husk will also be useful for steam generation which can be used in the process and the excess can be supplied to the nearby plants and generate some additional revenue.

## 3. MATERIALS AND METHOD

### 3.1 Materials

Wheat husk obtained from local milling companies as well as collected from local agricultural field were first of all washed thoroughly with deionized water to remove the adhering soil and dust particles. They were then dried under sunlight for 48 hours. The dried husks were grounded into powder and sieved through 20µm mesh size. All chemicals used were from local supplier and used as received.

### 3.2 Method

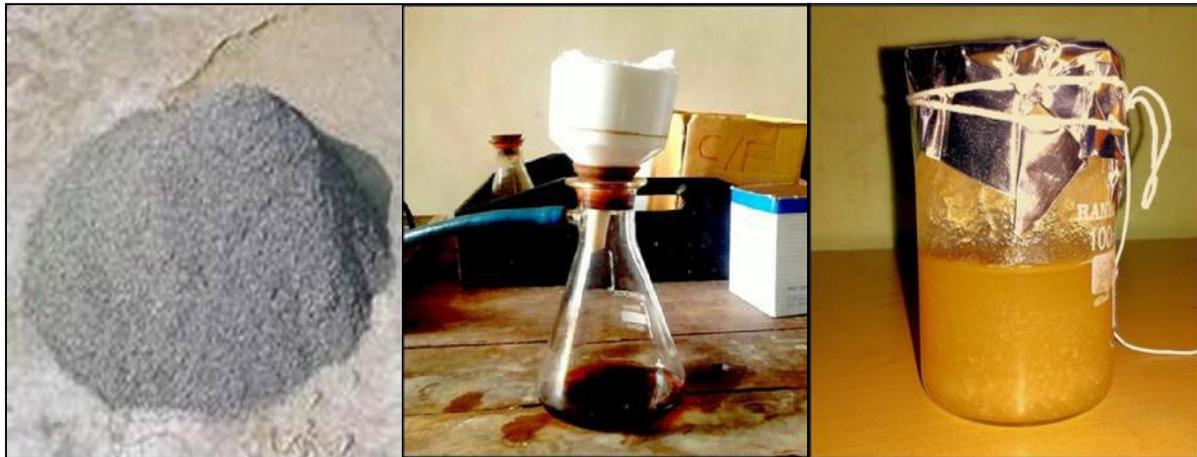
#### 3.2.1 Preparation of Rice Husk Ash:

Ash obtained by burning 100 gm. of Wheat husk in Muffle furnace at 500<sup>0</sup> C for 5 hrs because it is known that ashes with high silica content are obtained at temperatures lower than 400<sup>0</sup>C [Kamath and Proctor 1998][9,10]. Ozgul-Yucel et al. [2004][11] also demonstrated that rice husk ash has an amorphous structure when burned over 500<sup>0</sup>C. Thus 500<sup>0</sup>C was chosen as the burning temperature. The temperature in the Muffle

furnace monitored with the help digital temperature probe. Once the white ash is formed, it was weighed and found to be  $\frac{1}{4}$  of the mass of the Wheat husk i.e. 25 gm.

### 3.2.2 Preparation of Sodium Silicate from Rice Husk Ash:

Thus ash produced in the first step is mostly amorphous silica which is reactive around  $100^{\circ}\text{C}$  with NaOH solution at atmospheric pressure to yield sodium silicate. For the proper digestion of WHA in caustic, 3N solution of NaOH was used. The reaction time and the temperature for heating is 30 min at  $80^{\circ}\text{C}$ . A viscous, transparent, colorless sodium-silicate solution ( $\sim 15\%$  w/w) is obtained after filtration of the reacted slurry (consisting of residue digested ash, sodium silicate, water and free sodium hydroxide).



**Figure 3.1** Ash at  $350^{\circ}\text{C}$ , Sodium Silicate Solution and Precipitated Silica

### 3.2.3 Precipitation of Silica from Sodium Silicate solution:

The concentrated solution is the gelatinous sodium silicate taken in 200 ml beaker and concentrated Sulphuric acid ( $\text{H}_2\text{SO}_4$ ) added until solution becomes acidic. The addition of Sulphuric acid is done very slowly (otherwise the chemistry of such mass may change along with physical properties) until acidic conditions indicate approximately complete precipitation of silica from sodium silicate. Silica was then precipitated from the solution and sodium sulphate as a by-product remains at bottom. Little quantity of water  $15\text{-}20\text{ cm}^3$  is added to reduce high exothermic temperature. Solution was kept as it is for half an hour to cool. Solution was filtered by using filter paper no.41 by giving water wash. The temperature is in the range of  $80\text{-}90^{\circ}\text{C}$  and pressure is the normal atmospheric pressure. A white precipitate of silica in solution of sodium sulphate is obtained. The silica (wet impure silica) obtained above is filtered. Purification of this silica for removal of Sulphate impurities constitutes the third step of the process.

### 3.2.4 Determination for Rate constant:

For finding Rate constant of reaction, 5 ml of sample was taken out after each 5 min. 5 ml of 3N HCl was added to it and titrated against 3N NaOH, burette reading was noted down. Rate constant was calculated experimentally and graphically. The results were tabulated for different samples by varying reaction time and temperature in table 4.2 and 4.3. The residue cake of filter is then weighed & noted down. Clear filtrate obtained was aqueous sodium silicate solution and further concentrated in the oven by keeping temperature range between  $100\text{ to }150^{\circ}\text{C}$  for 60 to 90 min.

For reaction-1 in section 2.2, rate of disappearance of A is given by

$$-r_A = \frac{-dC_A}{dt} = KC_A^2C_B \quad (1)$$

For above equation, rate constant expression was obtained and given below as,

$$K = \frac{-l_i \left\{ \frac{2C_{B0} - C_{A0} + C_A}{2} \right\}}{(2C_{B0} - C_{A0})^2} + \frac{(2C_{A0}^2 - 1)l_i C_A}{(2C_{B0} - C_{A0})^2 (2C_{B0} C_{A0})} + \frac{2}{-(2C_{B0} - C_{A0})C_A} \quad (2)$$

For our convenience, we assumed  $Kt = Z$  where  $Z$  is the RHS from equation (2).

#### 4. RESULTS AND DISCUSSION

Table 4.1 shows results obtained for each sample in laboratory. It has been observed that at 55<sup>o</sup>C, the pH is very less than the actual pH value of silica. Further as we increased the temperature from 55<sup>o</sup>C to 90<sup>o</sup>C and varying the reaction time, the value of pH goes on increasing and at 80<sup>o</sup>C and 15 min reaction time, we get correct pH value of silica.

In case of specific gravity, as we go on changing temperatures there is not much deflection in values as compared to the actual value of specific gravity of silica. But at 80<sup>o</sup>C, we get correct value of specific gravity of silica.

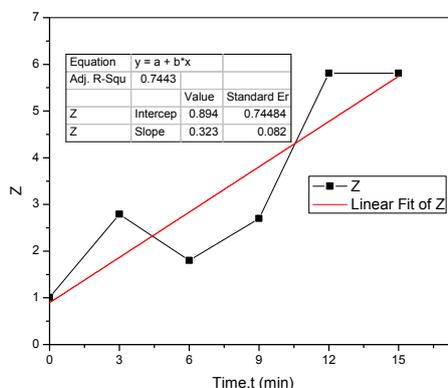
**Table 4.1 Results for pH and Specific Gravity at different Temperatures and Reaction Time**

Run No.	Sample	Reaction time	Temperature	pH	Specific Gravity
1	A	21	55	5.6	2.50
2	B	18	60	5.9	2.55
3	C	18	65	6.0	2.59
4	D	21	70	6.1	2.60
5	E	15	80	6.3	2.63
6	F	21	90	6.4	2.70

At a temperature of 80<sup>o</sup>C, the initial concentrations of reactant A ( $C_{A0}$ ) and B ( $C_{B0}$ ) were 2.30 and 2.50 gmol/lit respectively. The final concentrations obtained after 15 min reaction time for reactant A ( $C_A$ ) and B ( $C_B$ ) were 0.3 and 0.5 gmol/lit. The average value of rate constant is found to be 0.3195 (gmol/ lit)<sup>-1</sup>(min)<sup>-1</sup>. Slope of graph  $Z$  v/s Time at temperature 80<sup>o</sup>C is found to be 0.323, hence for experiments  $K$  graphically is 0.323 (gmol/lit).

**Table 4.2 Rate constant at 80<sup>o</sup>C**

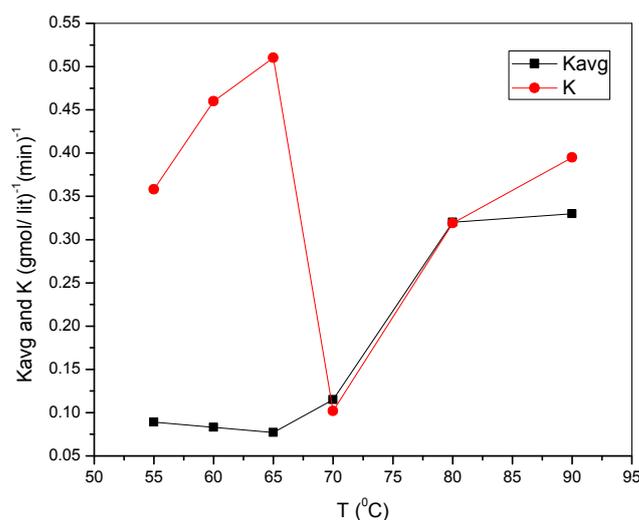
Sr. No.	Time, t (min)	Z	K, (gmol/lit) <sup>-1</sup> (min) <sup>-1</sup>
1	0	1.01	-
2	3	2.79	0.33
3	6	1.80	0.30
4	9	2.70	0.31
5	12	5.81	0.48
6	15	5.81	0.38



**Figure 4.1. Plot of Z v/s Time**

**Table 4.3 Rate constant for different temperatures and reaction time**

Sample	A	B	C	D	E	F
$K_{avg}$	0.089	0.083	0.077	0.115	0.320	0.33
$K(\text{gmol/lit})^{-1}(\text{min})^{-1}$	0.358	0.460	0.510	0.102	0.319	0.395



**Figure 4.2. Graph of  $K_{avg}$  and  $K$  vs. Temperature**

## 5.CONCLUSION

Wheat husk is not just a waste material but more than that. Thus instead of just disposing it off one can add value to it by effectively utilizing silica content present in it and to produce precipitated silica from it. From the experiments, it has been observed that the approximately 90% of  $\text{SiO}_2$  is recovered. The main advantage of this proposed process is that waste disposal problem of Wheat husk is eliminated and also valuable product can be obtained.

From experimental results,  $80^\circ\text{C}$  is found to be the optimum temperature as the quality of silica obtained was more pure and dry. Purity of Silica obtained from  $80^\circ\text{C}$  was 89.70 % (on dry basis). The rate constant determined experimentally and also graphically and found that the values of  $K_{avg}$  (experimentally) and  $K$  (graphically) are approximately equal at  $80^\circ\text{C}$  of reaction time 15 min whereas there is lot difference between these values at other temperatures and reaction time.

## REFERENCES

- [1] Khushbu G. Patel, Rakshith R. Shettigar, and Nirendra M. Misra, "Recent Advance in Silica Production Technologies from Agricultural Waste Stream–Review", Journal of Advanced Agricultural Technologies, Vol. 4, No. 3, September 2017, 274-279.
- [2] Khushbu G. Patel, Nirendra M. Misra, and Rakshith R. Shettigar, "Preparation and Characterization of Silica Gel from Wheat Straw", International Journal of Chemical Engineering and Applications, Vol. 7, No. 5, October 2016, 344-347.
- [3] K.Bogeshwaran, R.Kalaivani, Shifna Ashraf, G.N.Manikandan, George Edwin Prabhu, "Production of Silica from Rice husk", International Journal of ChemTech Research, Vol.6, No.9, September 2014, 4337-4345.
- [4] RaginiPatil, RajendraDongre and JyotsnaMeshram, "Preparation of Silica Powder from Rice Husk", IOSR Journal of Applied Chemistry (IOSR-JAC), 2014 (ICAET-2014), 26-29.
- [5] DavinderMittal, "Silica from Ash-A Valuable Product from Waste Material", Resonance – Journal of Science Education, Volume 2, Issue 7, July 1997, 64-66.

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- [6] Pinar Terziglu and SevilYucel, “Synthesis of Magnesium Silicate From Wheat Husk Ash: Effects of Parameters On Structural and Surface Properties” *BioResources*, Vol 7, No 4 (2012), 5435-5447.
- [7] S H Javed, Anam S Umair and Tajwar S, “Precipitated Silica From Wheat Husk”, *Journal of Pakistan Institute of Chemical Engineers*, Vol. XXXIX (2011), 51-54.
- [8] V R Shelke, S SBhagade and S A Mandavgane, “Mesoporous Silica from Wheat Husk Ash”, *Bulletin of Chemical Reaction Engineering & Catalysis*, 5(2), 2010, 63-67.
- [9] IsakRajjakShaikh and Alangir Abdulla Shaikh, “Utilization of Wheat Husk Ash as Silica Source for the Synthesis of MCM-41 Type Mesoporous Silicates: A Sustainable Approach towards Valorization of the Agricultural Waste Stream”, *Research Journal of Chemical Sciences*, Vol. 3(11), November (2013), 66-72.
- [10] Dodson J R, “Wheat Straw Ash and its Use As a Silica Source”, University of York, Chemistry, September 2011, PhD Thesis.
- [11] NittayaThuadaj and ApinonNuntiya, “Preparation of Nano silica Powder from Wheat Husk Ash by Precipitation Method”, *Chiang Mai Journal of Science*, 35(1), 2008, 206-211.