A
Dissertation Report
On

EXTRACTION OF ACTIVATED CARBON AND SILICA FROM RICE HUSK

Submitted in partial fulfillment of the requirement for the award of the degree of

MASTER OF SCIENCE
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Submitted by
PRIYANKA DEVI
(Reg. No.: 301602035)

Under the supervision of

Dr. Avinash Chandra
Associate Professor
Department of Chemical Engineering

THAPAR INSTITUTE
OF ENGINEERING & TECHNOLOGY
(Deemed to be University)

SCHOOL OF CHEMISTRY AND BIOCHEMISTRY
THAPAR INSTITUTE OF ENGINEERING & TECHNOLOGY
(Deemed to be University)
PATIALA-147004 (INDIA)

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CERTIFICATE

This is to certify that the dissertation entitled, **“EXTRACTION OF ACTIVATED CARBON AND SILICA FROM RICE HUSK”** being submitted by Ms. Priyanka Devi in partial fulfillment for the requirements for the award of degree of Masters of Science in chemistry to the School of Chemistry and Biochemistry, Thapar Institute of Engineering & Technology, Patiala is a bonafide work carried by her under my supervision. The contents of this dissertation have not been submitted for the award of any other degree or diploma.

Priyanka

Priyanka Devi

Date: 01-08-2018

This is to certify that the statement made by the candidate is correct and true to the best of our knowledge.

01/08/18

Dr. Avinash Chandra
Associate professor
Department of Chemical Engineering
Thapar Institute of Engineering & Technology, Patiala-147004

DECLARATION

I hereby declare that the work being presented in the dissertation entitled "**EXTRACTION OF ACTIVATED CARBON AND SILICA FROM RICE HUSK**" in the partial fulfillment of the requirements for the award of the Degree of Master of Science in Chemistry, and being submitted to the School of Chemistry and Biochemistry, Thapar Institute of Engineering & Technology Patiala. This is my own work during the period of January to July 2018, under the supervision of Dr. Avinash Chandra (Department of Chemical Engineering). I have not submitted the contents embodied in this dissertation for the award of any other degree.

Place: T.I.E.T, Patiala

Date: 01-08-018

Priyanka

Priyanka Devi

This is to certify that the statement made by the candidate is correct and true to the best of our knowledge.

01/08/18

Dr. Avinash Chandra
Associate professor
Department of Chemical Engineering
Thapar Institute of Engineering & Technology, Patiala-147004

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Priyanka

Priyanka Devi

ABSTRACT

The activated carbon and silica have been aiming to extract from rice husk to convert agro waste into useful product at various calcination temperatures and processing time. The obtained were analyzed by SEM, BET, EDS and XRD. The various properties of activated carbon and silica at all operating conditions were reported. These properties include surface area, pore volume and surface morphology, etc. The maximum 74.63% weight and 72% weight of activated carbon were obtained for the observation time 14 hours and 12 hours respectively. The processing time of 14 hours and the processing temperature as 100°C are optimized for activated carbon. The BET surface area, total pore volume and average pore size of the activated carbon are found as 4.683m^2/g, 8.886cm^3/g and 7.590nm respectively. For silica, the calcination temperatures were chosen as 400, 500, 600, 700 and 800 for the present work. The temperature 500°C and above were founded as optimum for obtaining the regular particle size. The EDS confirmed 100% silica as product at all the temperature and processing time. The XRD analysis of obtaining silica is shown that the silicon is also present along with silica. The BET surface area, total pore volume and average pore size of the silica are 280.68 m^2/g, 0.6553 cm^3/g and 9.339nm respectively.

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ABBREVIATION

N - Normality

RA - Residual Alkali

MB - Methylene Blue

RH - Rice Husk

RHA -Rice Husk Ash

GC -Gas Chromatography

MS - Mass Spectroscopy

XRF- X-Ray Fluorescence

BET-Brunauer Emmett Teller

FTIR - Fourier Transformation Infrared Spectroscopy

SEM - Scanning Electronic Microscopic

EDS- Electron Data System

XRD-X-ray Diffraction

TGA - Thermo Gravimetric Analysis

DSC - Differential Scanning Calorimetry

ICP –Inductively Coupled Plasma

MS- Mass Chromatography

PEG- Polyethylene Glycol or Percutaneous Endoscopic Gastrostomy

MG - Malachite Green

MAS - Magic Angle Spinning

EA- Electron Affinity

TEM- Transmissionelectron microscopy

CHAPTER-1 INTRODUCTION

The Agra waste is abundantly available in our country being an agricultural dominating country. A little part of this waste is usually used as cattle feed, electricity generation, bulking agents for composting of animal manure, boilers, production of biomass and household applications, etc. [1, 4]. A large fraction of these agro waste are burned at the field itself, which leads lot of environmental pollution and green house gases generation. Due to the burning of agro waste at the field, the fertility of the field get reduced, which are not desirable at all. Hence, the effective use of agro waste are required for the betterment of our society and planet.

The rice husk is one of the agro waste that found abundantly throughout the country. It is hard protective coverings of grains of rice, are generated in large amount of agro waste material in the rice production areas of the world. Asia is the one of the major producer of rice.The major rice producing countries are listed inTable 1.1.

Table1.1: World rice husk production[3, 4]

S.No.	Country	Rice paddy tonne	Rice husk tonne
1.	Bangladesh	27	5.4
2.	Brazil	9	1.8
3.	Burma	13	2.6
4.	China	180	36
5.	India	110	22
6.	Indonesia	45	9
7.	Japan	13	2.6
8.	Korea	9	1.8
9.	Philippines	9	1.8
10.	Taiwan	14	2.8
11.	Thailand	20	4
12.	US	7	1.4
13.	Vietnam	18	3.6

China and India are collectively accounted for more than one half of the world's rice supply, In Tamil Nadu India, is the third ranking state in the production of paddy after West Bengal and Andhra Pradesh. Paddy production is nearly 7 million tones in Tamil Nadu [5,6]. A representative picture of rice paddy is shown in Figure 1.2. The rice paddy consists about 20-22% of husk [7]. The silica content ishighinrice among other agricultural waste. Due to heigh silica content, the rice husk is not used in paper and pulp production. The one tone of rice paddy produces 20kg of rice husk (20%). The quality of rice husk ash depends upon the variety of rice paddy,cultivation conditions and geographical location [7,3].

Figure 1.1: Dried rice paddy

The farm fresh rice is coveredwith fibrous hard brown covering. This outermost covering of rice is termed as rice husk and it contained silica[8]. Rice husk is woody in nature and insoluble in water [8]. The rice husk is also the by-product of rice industry. The rice husk production in India is 120 million tonnes per year.

The rice husk is having similar chemical composition as numerous common organic fibers having such as cotton, hemp,sisal, jute and bagasse etc. [9, 10]. Table 1.2 shows the average composition of rice husk produces in Asia and western world. It has cellulose 38%, ash 20%, pentosans 18%, lignin 22%, D-xylose 17.52%,α-cellulose 43.30%, l-arabinose ($C_5H_{10}O_5$)6.53%, D-galactose 2.37%, Methylglucuronic acid 3.27%and hemicellulose 32.7% [11, 12].

Table 1.2 : Major constituents of rice husk[11-12]

S.No	Constituents	Percentage
1.	D-xylose	17.52%
2.	Ligin	22%
3.	l-arabinose	6.53%
4.	Methylglucuronic acid	3.27%
5.	D-galactose	2.37%
6.	α-cellulose	43.3
7.	Pentosans	18%
8.	Hemicellulose	32.70%
9.	Ash	20%
10.	Cellulose	38%

The rice husk also contains Al_2O_3(0.82%), carbon 66.67%, Na_2O is 0.78%, Fe_2O_3 is 0.78%, mass% of MgO is 0.21, SiO_2 is 22.3%, CaO is 0.24%, K_2O is 1.10% and H_2O is 7.1%[13].Therice huskcomposition is depends on the various factors as its variety, geographical conditions, climatic variation, soil type, fertilizer used, agronomic practices and timeliness of crop[14,15]. The components present in rice husk are listed in Table 1.3.The calorific value of rice husk is 12.1-15.2MJ/kg and average moisture content is 8.0-9.0%. The physical properties of rice husk along with elemental percentage are given in table 1.4 [8, 16].

Table 1.3: Chemical components analyse in rice husk[13]

S.No	**Components**	**Weight %**
1.	Aluminum oxide(Al_2O_3),	0.82
2.	Carbon (C)	66.7
3.	Sodium oxide (Na_2O),	0.78
4.	Iron oxide(Fe_2O_3),	0.42
5.	Magnesium oxide (MgO),	0.21
6.	Silica (SiO_2)	22.3
7.	Calcium oxide (CaO),	0.24
8.	Potassium oxide (K_2O),	1.1
9.	Water (H20)	7.1

There are various reserach has been carried out on the use of rice husk [1- 16]. The rice husk is useful in a various application which includes production of bioethanol [17], fermentable sugar [18], Lightweight bricks [19, 20]. It has high hemicelluloses and cellulose content that can be easily hydrolyzed into fermentable sugars [17, 18], Lightweight bricks [19], particle board [21], industrial adsorbants [22], fuel for domestic and industrial applications [23], fertilizer production [24], dye [25], filtration of arsenic from water [26] etc. The lightweight bricks were made from different percentage of rice husk ash with clay sand [20]. These bricks and particle board [21] are widely used for interior decoration, construction of lightweight furniture [20]. The rice husk ash has better adsorptive property and has been used to be prominent for adsorptive (waste water treatment) removal of metal ions (organic or inorganic compounds), used as cleaning of atmosphere contaminants (methane, carbon dioxide etc.) refining of gold thiourea complex [27, 8], and The role offermented sugar was in digestive system, polyols, monosaccharides, which include oligosaccharides, and disaccharides. Fermentable sugar were obtained from different conditionhydrolysates the rice husk and evolved in ethanol [28].

Table 1.4: Properties of rice husk ash[8, 16]

S.No.	Property	Range
1.	Bulk density (kg/m3)	9-160
2.	Length of husk (mm)	2-5
3.	Hardness (Mohr’s scale)	5-6
4.	Ash (%)	20-29
5.	Carbon (%)	35
6.	Hydrogen (%)	4-5
7.	Oxygen (%)	31-37
8.	Nitrogen (%)	0.23-0.32
9.	Sulfur (%)	0.04-0.08
10.	Moisture (%)	8-9

Hence, the present work is aiming the production of charcoal (activated carbon) and extraction of elemental silica. The charcoal and silica are obtained at various process conditions and characterised.

Zhang *et al.* (1996)[44]studied the role of rice husk ash in concrete on the hydration, microstructure of aggregate and cement paste. The calcium hydroxide and calcium silicate hydrates was found in Portland cement paste that is used in rice husk ash paste and its hydration. The main conclusion drawn from the study was that the Portland cement paste increased the compressive strength as compare to cement paste.

Kalapathy *et al.* (2000)[32]studied that pure silica xerogels were obtained from rice husk ash. The silica gels were produced from rice husk ash heated to 80°C for 12 h to obtain xerogels. Other mineral contents and silica were determined by (EDX), energy dispersive X-ray and (ICP) inductively-coupled plasma emission spectrometers, respectively. The xerogels produced from rice husk having 93% silica, 2.6% moisture and 4.4 % impurities (Na, K and Ca).

Adam *et al.* (2002)[49]concluded that silica gels and powders have been widely used in industry. Thesilica powder was used asadsorbents, fillers, and catalyst supportsfor various industrial catalysts etc. The sol-gel technique was used to prepare catalyst for industrial waste water treatment using rice husk.

Chandrasekhar *et al.* (2003)[11]reported that the rice husk can be used as a fuel for direct fired furnaces. They reported that rice husk was contained 20% silica in hydrated amorphous form with high reactivity, large surface area and ultra fine particle size under controlled temperature conditions. This silica was used for preparing SiC, Si_3N_4, elemental Si, Mg_2Si, etc.

Liou (2004)[45]have used rice husk to extractedsilica and activated carbonusing non-isothermal conditions. This study was based upon physical, chemical and morphological characteristics of silica and activated carbon extracted from rice husk ash such as Scanning electronic microscopic (SEM), Fourier transformation infrared spectroscopy (FTIR), Electron Affinity (EA), X-ray Diffraction (XRD), ICP-MS andBET. The average pore diameters silica and activated carbon (AC) were 2.2 and 5.4 nm and surface area were 261 and 235 m^2/g respectively. It was concluded that the increase in pore diameter and pore volume of silica and activated carbon with decrease in rice husk heating rate.

Tang *et al.* (2005)[41]haveproduced silica aerogel from rice husk ash (RHA) by frequent critical carbon dioxide drying. The characterization of silica was done using Transmission electron microscopy, Brunauer Emmett Teller, and scanning electron microscopy. They found BET surface area as of $597.7m^2/g$ and pore volume as $8.65cm^3/g$.

Rahman *et al.* (2005)[29]studied that activated carbon was obtained by acid leaching of rice husk ash. The obtained activated carbon was used in absorption of malachite green (MG). The rice husk ash was obtained at 500°C for better adsorption capacity. They observed that the activated carbon obtained from rice hush had excellent adsorption capacity.

Nair *et al.* (2007)[43]determined that the structure of the rice husk ash by pozzolanic activity (degree of reaction measure in over time).The highest % of silica was obtained by the pozzolanic material of rice husk ash at 500°C to 700°C in 12 hours. The characterization was done using conductivity measurement, (MAS) magic angle spinning, chemical analysis, microscopic, X-ray diffraction and nuclear magnetic resonance.

Thuadaij *et al.* (2008)[33]were obtained pure silica and nano silica by the precipitation method at 700°C. The pure silica was obtained from rice husk ash (RHA) using activating agent as sodium hydroxide, sulphuric acid (pH - 2) and ammonium hydroxide (pH < 9).The obtained silica jelly was dried for 12h. The nano silica was obtained from rice husk ash refluxing with HCl (6N) for 4hours. The obtained forms of silica were characterized by FTIR (Fourier transfer infrared spectroscopy) and X-ray florescence.

Kalderis *et al.* (2008)[37]had obtainedactivated carbon from rice bagasse or rice husk using $ZnCl_2$, NaOH and H_2SO_4. The highest surface area was obtained for activated carbon using Brunauer Emmett Teller (BET) analysis.

Salas*et al.* (2009)[53] explained the rice husk ash had used in high performance concrete. The amorphous silica was present in rice husk ash with highly reactivity. The flexural and compressive strengths of silica fume concrete were comparable to rice husk ash concrete.The strengths of rice husk ash mixtures (chemically treated) is less than rice husk ash and silica fume concrete. They observed that the high performance concretes of rice hush ash had excellent used as supplementary cementing material.

Pankaj *et al.* (2010)[52]explained the method to remove methylene blue from waste water using rice husk ash at neutral pH and 596°C temperature. This study was indicated the adsorption of dyes was spontaneous and endothermic. It may be used in treatment of undesirable species in waste water and other harmful in effluents.

Adam *et al.* (2010)[39] haveused rice husk to extract silica-tin nanotubes by using sol gel method. The characterization of obtained silica-tin nanotubes was done using nitrogen adsorption-desorption isotherm, X-ray Diffraction, Fourier transformation infrared spectroscopy. The obtained silica-tin nanotubes were used for the ejectment of methylene blue via photo catalytic activity.

An *et al.* (2010)[46]extracted silica powder using carbonation route from rice husk ash. The yield of silica obtained is 72.52% and the characterization of silica has been done by inductively coupled plasma (ICP), infrared spectra, X-ray diffraction and emission spectroscopy

Lung *et al.* (2011)[31]reported the 20% increase in compressive strength of concretes using rice husk ash. The obtained rice husk ash has been concreting with better or comparable properties than without rice husk ash.

An *et al.* (2011)[47] **e**xtracted silica powder and activated carbon from leaching of rice husk ash using Na_2CO_3 which is also known as consecutive method. The 84.57% silica was obtained by base treated rice husk ash. The remaining solid residue is basically the activated carbon which had good adsorption capacity. The characterization was done by X-ray Diffraction, atomic emission measurement, Fourier transformation infrared spectroscopy, scanning electrode microscopic and Brunauer Emmett Teller.

Rafiee *et al.* (2012)[42] explained the thermal combustion to extract silica from rice husk. The 90.469% silica was extracted from rice husk ash under controlled conditions. They was optimized the conditions to obtained highly pure silica, a nano sized, highly reactivity, 99.9% in amorphous form and large surface area.This nanosilica was showed large number of acidic sites and strong in similitude with commercial silica that is making for catalysts. The characterization of nanosilica has been done by Brunauer Emmett Teller, nitrogen adsorption desorption isotherm, X-Ray Fluorescence, Transmission electron microscopy, and X-ray Diffraction. The surface area was observed $623m^2/g$ and total pore volume is $0.02408cm^3/g$.

Manique *et al.* (2012)[35]explained the use of rice husk ash for purifying biodiesel from waste frying oil at different concentration. The waste frying oil was shown the capability to adsorb the inorganic and organic impurities of biodiesel. The fine concentration was observed 4% for expulsion of the impurities from biodiesel. The silica was obtained from rice husk ash and it is having high adsorption capacity that is confirmed by Electron data system and Fourier transformation infrared spectroscopy analysis. Hence, the rice husk ash may use for the treatment of an agro-industrial waste pollutant.

Ghosh *et al.* (2013)[51] was extracted silica and activated carbon from rice husk at different time interval of calcination. The silica and activated carbon was extracted at 850°C and 900°C. The activated carbon was used for wastewater treatment.

Lin *et al.* (2013)[48] obtained mesoporous activated carbon for excellent adsorption capacity to remove methylene blue in aqoues solutions. The mesoporous activated carbon was prepared from alkali treated rice husk ash residue. The adsorption procedure was endothermic and spontaneous that is indicated by pseudo-second -order model and Langmuir isotherm dynamic.

Adam *et al.* (2013)[38] explained the utilization of tin and titanium incorporate rice husk. The silica was applied for removal of methylene blue as adsorbent. The silica has been characterized by BET, X-ray. Adsorption rate of the methylene blue is depended on pH of the solution. The BET surface area of silica is 315m^2/g and total pore volume is 0.367cm^3/g.

Cheenmatchaya *et al.* (2014)[34]extracted activated carbon by using the gasoline adsorption from acid leached rice husk ash. The rice husk was carbonized at 450°C for 2 hours. The activated carbon was used as adsorbent for waste water treatment. The BET surface area of the activated carbon was obtained from rice husk ash is 336.35 m^2/g.

Van *et al.* (2014)[50]was obtained the activated carbon (AC) from rice husk (RH) by using activation sodium hydroxide at diffèrent temperature. The Characterization has been done with the help of nitrogen adsorption desorption isotherm, Thermo gravimetric analysis (TGA-DTA), Scanning electronic microscopic (SEM) and Fourier transformation infrared spectroscopy (FTIR). They found maximum specific surface area at 800°C.

Hieu*et al.* (2015)[30] have usedVietnam rice husk to extract activated carbon. The silica was also separated before activation of rice husk ash. The silica was separated up to 95.6% from the rice husk ash. The activated carbon was used for adsorption of waste gases present in various plants, wastewater treatment processes.

Teo *et al.* (2016)[36]obtained activated carbon at different temperatures from rice husk. They had used supercapacitor electrode for evaluating their performance such as porosity and surface area at 8°C. They concluded the activated carbon is potentially suitable material for using supercapacitor electrode.

Bakar *et al.* (2016)[40]produced high pure silica from acid leached or unleached rice husk at 600°C. The pure silica was characterization at different conditions such as BET, SEM and XRF. The surface area for un-leached silica, leached silica with Hydrochloric acid and leached silica with sulphuric acid were 116m2/g, 218m^2/g and 208m^2/g respectively. The pore volume of un-leached silica, leached silica with Hydrochloric acid and leached silica with sulphuric acid were 0.23cm^3/g 0.32cm^3/g and 0.31cm^3/g respectively.

Todkar *et al.* (2016)[54]explained the use of silica for waste water disposal. They recovered more than 90 percent silica.

Additionally, the obtained 20%silica by various researchers is tabulated in Table 2.1. The table 2.1 shows that the minimum % silica was obtained by Adam *et al.* (2002) [49] and the maximum % silica obtained as 99.98% by Riveros et.al (1986) [57]using various methods. Recently, Bakar *et al.* (2016) [40] had obtained 99% silica from rice husk ash, which is less than the %silica obtained by Zhang et al. (2010) [56] and Riveros et.al (1986) [57].

Table 2.1: Silica yield reported in literature

S.No.	**Silica%**	**References**
1.	93	Kalapathy *et al.* (2000) [32]
2.	20	Adam *et al.* (2002) [49]
3.	95	Della et al. (2002) [55]
4.	72.52	An *et al.* (2010) [46]
5.	84.57	An *et al.* (2011) [47]
6.	90.46	Rafiee *et al.* (2012) [42]
7.	99	Bakar *et al.* (2016) [40]
8.	90.11	Thuadaij *et al.* (2008) [33]
9.	99.87	Zhang et al. (2010) [56]
10.	99.98	Riveros et.al (1986) [57]

The BET surface area reported in the literature is summarized in Table 2.2. the maximum surface area is obtained byRafiee *et al.* (2012) [42] as 623m^2/g with pore volume 0.02408cm^3/g. the maximum pore volume was obtained by Tang *et al.* (2005) [41] with BET surface area 597.7m^2/g. the observation of Table 2.2 shows that the surface area and the pore volume of silica are depends upon the temperature and preparation method.

Table 2.2: BET surface area and pore volume of silica reported in literature

S.No.	BET specific surface area (m^2/g)	Total Pore volume (cm^3/g)	References
1.	315	0.367	Adam *et al.* (2002) [49]
2.	235	-	Liou (2004) [45]
3.	597.7	8.65	Tang *et al.* (2005) [41]
4.	623	0.02408	Rafiee *et al.* (2012) [42]
5.	116	0.23	Bakar *et al.* (2016) [40]
6.	218	0.32	
7.	208	0.31	

The various types of pore volume and BET surface area have been reported in the literature and the relevant studies are summarized in Table 2.3. Specifically, the BET total pore volume, surface area, meso pore volume, average pore size and micro pore volume are given in Table 2.3.The minimum BET surface area of 91m^2/g was obtained by Kalderis *et al.* (2008) [37] and maximum 2696m^2/g surface area was obtained by Teo *et al.* (2016) [36] at 600°C and 800°C respectively. The minimum 0.26cm^3/g total pore volume was obtained by Lin *et al.* (2013) [48] and Teo *et al.* (2016) [36] had obtained maximum total pore volume as 1.496 cm^3/g. The minimum 0.15 meso pore volume was obtained by Lin *et al.* (2013) [48] at 600°C and maximum 1.056 meso pore volume obtained at 950°C by Teo *et al.* (2016) [36]. The minimum 2.2nm average pore size was obtained by Liou (2004) [45]and maximum 4.706 nm average pore size obtained by Teo *et al.* (2016) [36].

Table 2.3: Literature review for BET analysis of activated carbon for different temperatures

S.No	Temp. (°C)	BET surface area (m^2/g)	Total pore volume (cm^3/g)	Micro Pore volume (cm^3/g)	Meso Pore volume (cm^3/g)	Average pore size (nm)	References
1.	750	2121	1.022	0.589	0.243	4.387	Teo *et al.* (2016) [36]
2.	850	2696	1.496	0.274	0.691	4.104	
3.	950	1592	1.293	0.052	1.056	4.706	
4.	600	406.7	0.26	114.5	0.15	3.05	Lin *et al.* (2013) [48]
5.	700	815.6	0.58	-	0.58	3.29	
6.	800	1015	0.75	-	0.75	3.42	
7.	850	728.3	0.68	-	0.68	3.85	
8.	450	336.35	-	-	-	-	Cheenmatchaya *et al.* (2014) [34]
9.	200	1936.62	1.22	-	-	-	An *et al.* (2011) [47]
10.	650	2520	1.2929	0.9637	0.3292	-	Van *et al.* (2014) [50]
11.	700	2482	1.2929	0.9453	0.3476	-	
12.	750	2617	1.3398	1.0081	0.3317	-	
13.	800	2681	1.4016	1.011	0.3906	-	
14.	600	91	-	-	-	-	Kalderis *et al.* (2008) [37]
15.	-	261		-	-	2.2	Liou (2004) [45]

Overall the literature suggest that there are further scope for producing silica and activated carbon from rice husk for various industrial application and catalytic loading. In the present work we are aiming to see the effect of temperature variation on extracted silica and activated carbon properties.

CHAPTER-3 MATERIAL AND METHOD

The chapter discusses the materials (properties and specifications) and methods used for the extraction of silica and activated carbon from rice husk (RH).

3.1 Materials

The dried rice husk (hard protecting covering of rice) obtained from different regions of Punjab, Haryana and Chandigarh (Figure 3.1.1). The inorganic crucibles and required chemicals are purchased from local market of Patiala. Crucibles are composed of clay-graphite, and carbon bonded silicon-carbide (refractive material) which can withstand up to temperature 900°C.

The chemicals (analytical grade) used in the extraction process of silica and activated carbons are as follows:

i. Sodium hydroxide
ii. Sulphuric acid
iii. Ammonium hydroxide
iv. Distilled water

The required equipment’s are as follows:

i. Muffle Furnace
ii. Buchner Funnel with filtration assembly
iii. Vacuum pump
iv. Centrifuge
v. Titration Assembly

vi. pH meter

vii. Electric oven

viii. Weighing balance

Figure 3.1: Dried rice husk

3.2 Method for extraction silica and activated carbon from rice husk (RH)

The rice husk was washed using warm water to remove the dirt. The washed rice husk was dried in the presence of sun light.The crucible weight was taken by using weighing balance and rice husk was put into the crucible and weight of the rice husk was taken by subtracting the weight of crucible. The crucible containing rice husk placed inside the muffle furnace at constant temperature for constant time. The obtained sample was cooled for 20 minutes at room temperature. The rice husk ash (RHA) was obtained and RHA yield was calculated using following relation

$$\text{RHA\%} = \frac{\text{Weight of ash (g)}}{\text{Weight of sample (g)}} \times 100$$

3.2.1 Methods for extraction silica from rice husk ash (RHA)

A schematic flow chart for preparation of silica from rice husk ash is shown in Figure 3.2. The carbonized rice husk ash (RHA) was treated with sodium hydroxide aqueous and the mixture was heated at elevated temperature. Then we obtained sodium silicate with aqueous solution. The solution was cooled at room temperature and mixture was filtered. The filtrate was titrated with sulphuric acid and ammonium hydroxide to obtained silica. The reaction is as follows:

$$SiO_2 \text{ (Ash)} + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$

The silica particles were generated from the solution by adding sulphuric acid as catalyst. The precipitate of silica gel is obtained according to following reaction:

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$$

The wet gel was then dried in oven at 100°C for some times. Silica was obtained from sodium silicate solution using a sol-gel technique. The white powder of silica was obtained. The silica percent was calculated by using the formula.

Formula used to calculate silica percent:

$$\text{Silica\%} = \frac{\text{Silica obtained (g)}}{\text{Weight of sample (g)}} \times 100$$

3.2.2 Method for extraction activated carbon (AC) from rice husk ash (RHA)

The Residue obtained from the mixture of rice husk ash and sodium hydroxide is used to extract activated carbon. The dried residues were crushed by using motor pistil. The solid residue was dried in oven for some time. The percentage of activated carbon extract from rice husk (RH) was calculated following relation.

$$\text{Activated carbon\%} = \frac{\text{Activated Carbon obtained (g)}}{\text{Weight of sample (g)}} \times 100$$

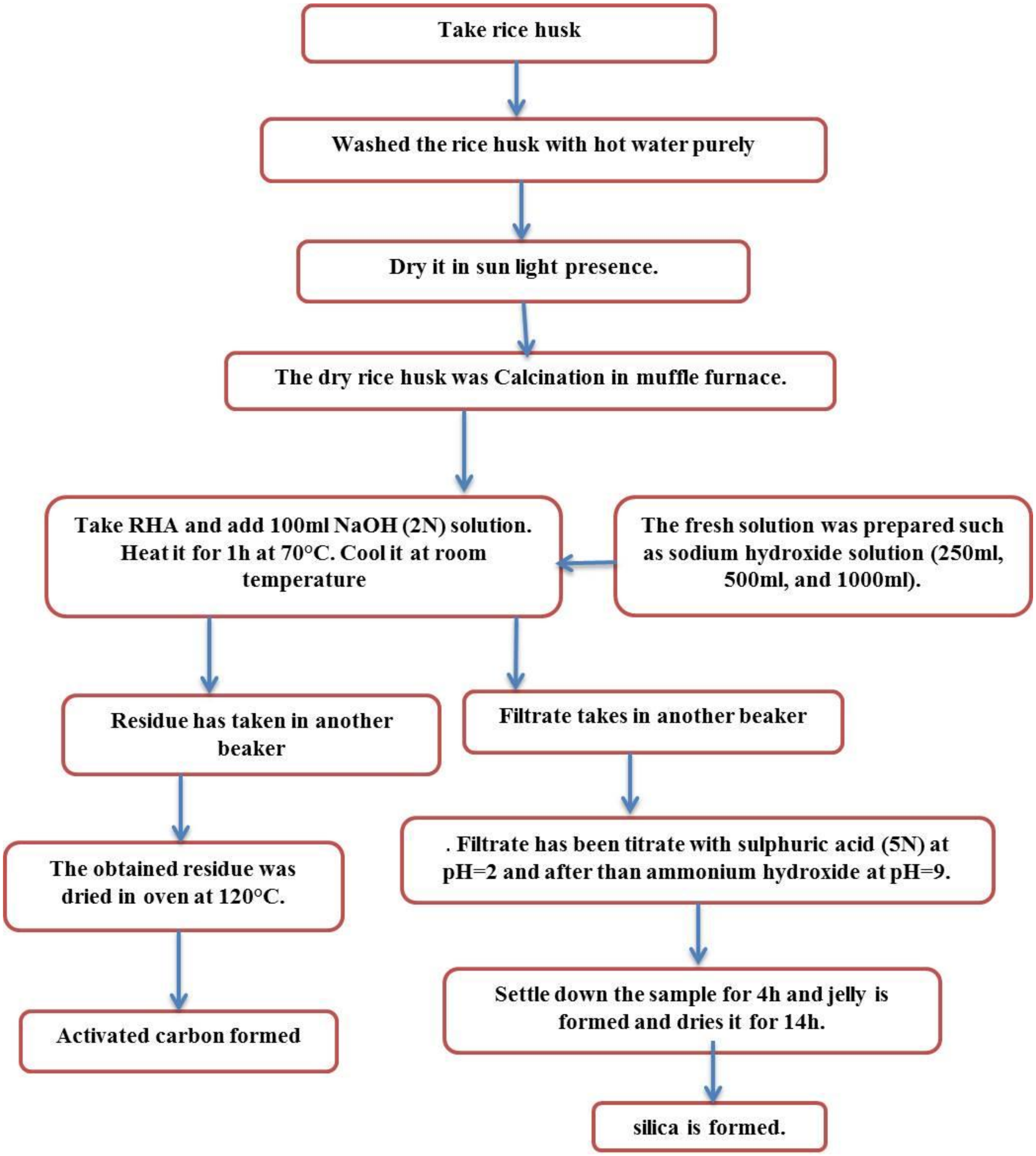

Figure 3.2: Flow diagram for preparation of silica and activated carbon

3.3 Chracterization

SEM

Surface morphology and grain size of the silica and activated carbon are analysed by scanning electron microscopy with a field-emission-scanning electron microscope (JEOL JSM-6510LV), operating accelerating voltage 15kV with magnification 1000 - 2500. Surface of the sample were coated with a thin layer of gold (3.5 nm) by the vacuum evaporation technique to minimize sample charging effects due to the electron beam.

X-ray diffraction (XRD)

The X-ray diffraction (XRD) were obtained X'Pert PRO Analytical diffractometer using Cu Kα radiation (k =1.540 A °) at 30kv and scan with the Bragg's angle (2h) in the range of 10–90 degree. The origin software was used to analyse the structural pattern of the sample.

Brunauer-Emmett-Teller (BET)

The surface area (m^2/g), total pore volume (Ml/g) and pore radius (nm) were calculated from the linear part of the Brunauer-Emmett-Teller (BET). Low-temperature nitrogen adsorption experiments were performed using a Quantachrome instrument
(Model Nova 2000, Quantachrome Instruments, Boynton Beach, FL, USA) system

Energydispersivespectrometer (EDS)

The elemental composition of the obtained material were analysed by EDS. The energy dispersive spectrometer was obtained OXFORD INCAx-act Analytical.

CHAPTER-4 RESULTS

The activated carbon and silica is being separate from rice husk. The rice husk has been converted (carbonized) in to rice husk ash at various temperature using electric furnace. The temperature condition has been chosen as 400°C, 500°C, 600°C, 700°C and 800°C. The carbonization time is also taken as 1 hour to 6 hours for containing rice husk ash (RHA). Collectively, the carbonising temperature and carbonizing time are the influencing variable for obtaining rice husk ash. The obtained RHA is being used to separate activated carbon and silica. The obtained RHA is shown in Figure 4.1.

Figure 4.1 : Rice husk Ash (RHA)

4.1 Analysis of Activated Carbon

TheActivated carbon has been obtained as per the procedure given in Chapter -2 (Material and Method). The obtained Activated Carbon shown in Figure 4.2. The obtained activated carbon has been analysed using SEM, EDS and BET.

Figure 4.2 : Activated Carbon

4.1.1 Scanning Electron microscopy (SEM)

The SEM images of obtained activated carbon are shown in Figure 4.3 at x1000, x2000, x2500 and x500 resolution**.** The Figure 4.3-A, C, E and G are the activated carbon obtained after 12 hours, where as the Figure 4.3- B, D, F and H are for the activated carbon obtained at procedure time of 14 hours.

The observation Figure 4.3.A and Figure 4.4.B shows that the process structure are obtained for both the areas but the density is higher in case of 14 hours with larger grain size than Figure 4.3.A. further at higher zoom of x2000 is same thing clearly visible at larger more size. The same thing is also confirmed with further Figure 4.3.D, F and H. Hence the activated carbon obtained at processing time 14 hours is qualitatively good with larger pore size , regular structure and higher pore density.

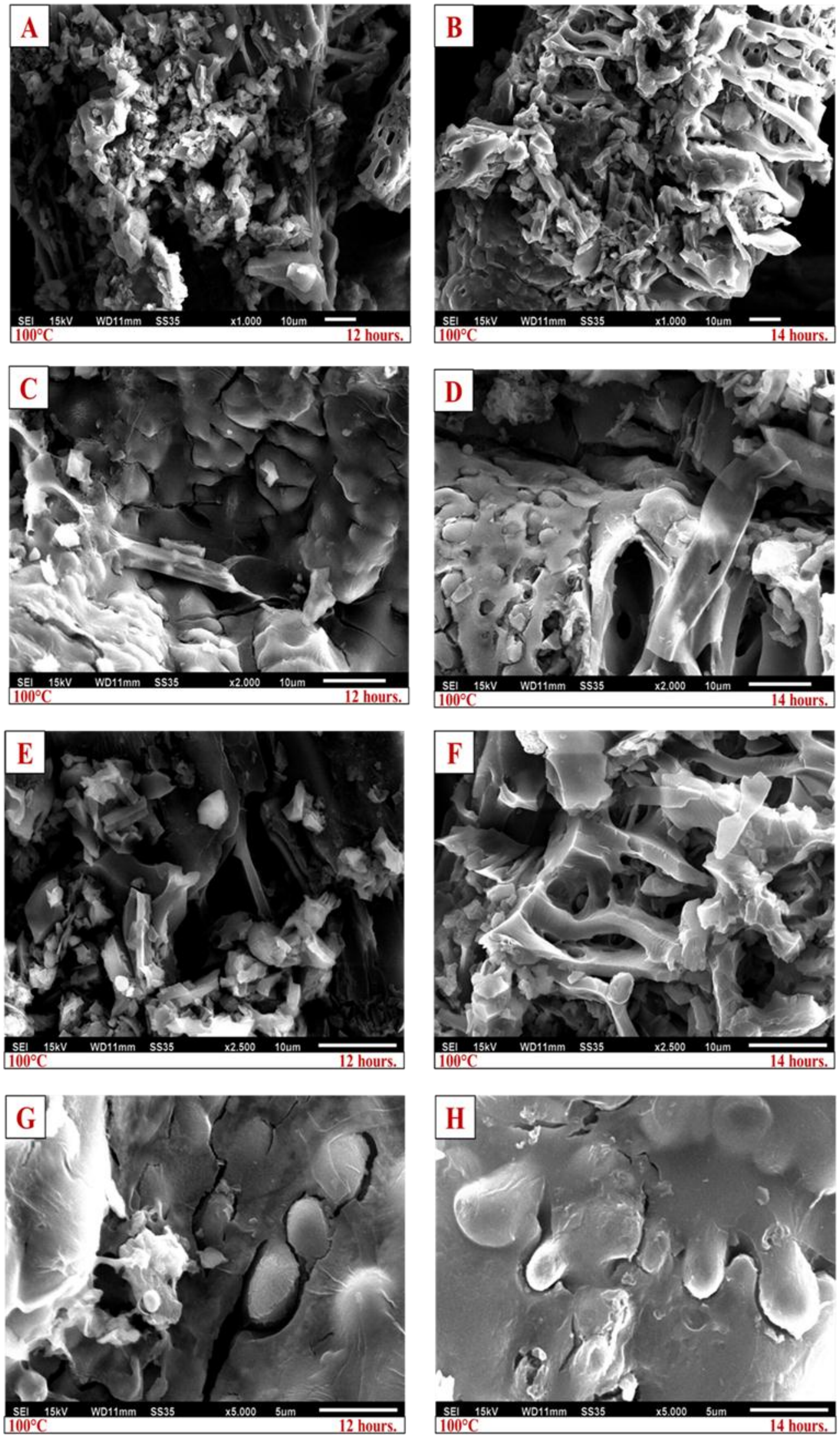

Figure 4.3 : SEM of activated carbon (A), 12h; x1000 (B), 14h; x1000(C), 12h; x2000 (D), 14h; x2000 (E), 12h; x2500 (F), 14h; x2500 (G), 12h; x5000 (H), 14h; x5000

4.1.2 Energy-dispersive X-ray spectroscopy (EDS)

The EDS images of activated carbon are shown in Figure 4.4. The figure 4.4.A shows the 72.07% activated carbon, 24% oxygen and 24.32% sodium. The maximum activated carbon obtained 72% for the observation time 12 hours at 100°C. In case of optimisations 14 hours the maximum activated carbon shown in Figure 4.4.B obtained as 74.63%, oxygen 22.32% and sodium 3.05% are shown in Table 4.1. In some samples a little amount of silica is also assume. The EDS images confirmed the activated carbon with almost 25% impurities. Their impurities can be minimise by taking are in the processing and proper separation of process reagents such as sodium hydroxide in case of activated carbon.

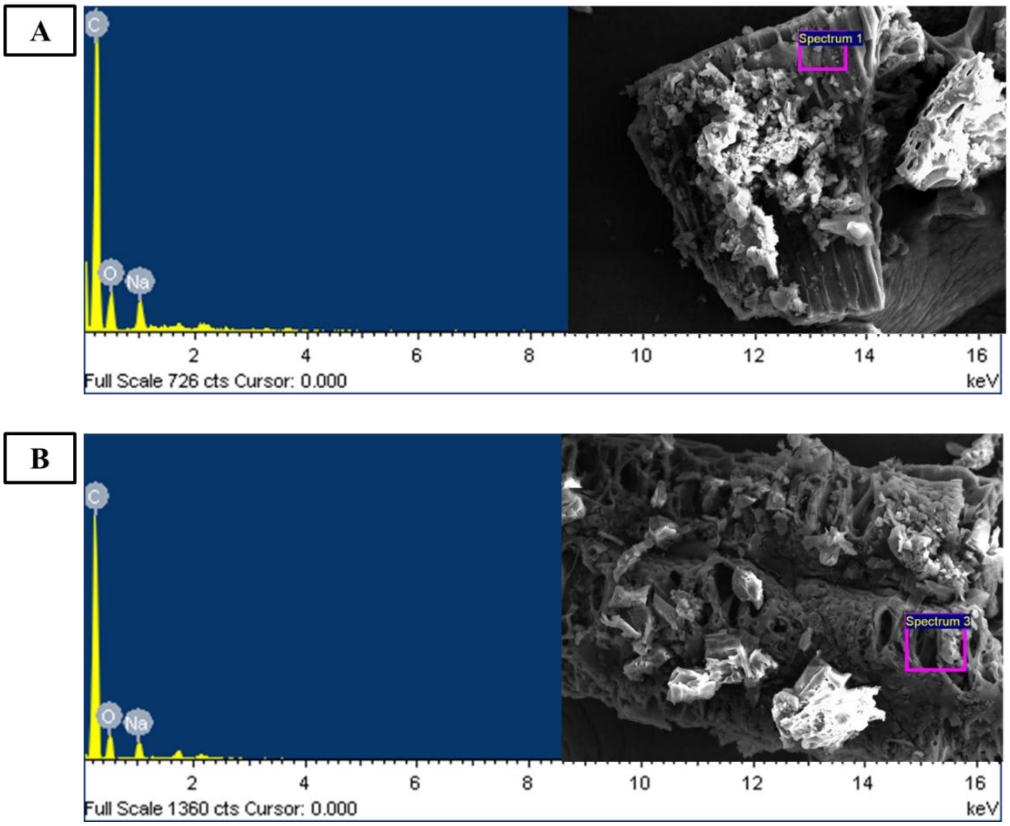

Figure 4.4 :EDS of activated carbon (A) 12h and (B) 14h)

Table 4.1: weight % obtained by EDS of activated carbon

S.No.	Temp.(°C)	Time (hour)	Carbon%	Sodium%	Oxygen%
1	100	12	72.07	3.61	24.32
2	100	14	74.63	3.05	22.32

4.1.3 Brunauer-Emmett-Teller (BET)

The BET of obtained silica has been performed to investigate the surface area and pore size. The result obtained BET is shown in the Table 4.2. The maximum BET surfaces has been obtained 4.6831m^2/gfor 14 hours processing and minimum 2.5841m^2/g for 14 hours processing. The maximum pore volume obtained 8.886cm^3/g for 14 hours processing time and minimum pore volume 6.621cm^3/g for 12 hours processing.

Table 4.2: BET surface area of activated carbon obtained at different time

S.No	Temp. (°C)	Time (hours)	BET specific surface area (m^2/g)	Total Pore volume (cm^3/g) x 10^{-3}	Average pore size (nm)
1	100	12	2.584	6.221	9.629
2		14	4.683	8.886	7.590

4.2 Analysis of Silica

The carbonized rice husk ash was used to prepared silica. The obtained silica was formed in very light, white porous powder form is shown in Figure 4.5 and the silica (weight %) was obtained on different carbonization time duration of rice husk shows in Table 4.2

Figure 4.5: Obtained silica after jelly dried at 100°C.

The Extraction of silica and activated carbon from rice husk ash. The rice husk sample after burning out at 400,500,600,700,and 800 for different time (1h to 6h) presented the amount of rice husk ash and silica shown in % Table 4.3. Took 5g of rice husk in crucible after burning found the amount of rice ash and treating with sodium hydroxide(2N), sulphuric acid(5N) and ammonium hydroxide to obtained silica at 400°C is ash 50 % and silica 46.4 % obtained in one hour, ash46.6 % and silica 33.4 % obtained in two hour, ash 38.2 % and silica 50 % obtained in three hour, ash 32.6 % and silica 48 % obtained in four hour, ash 32.2 % and silica 40 % obtained in five hour and ash 32 %, silica 42 % obtained in six hour.

The amount of rice husk ash and silica obtained in 500°C is ash 13.52 % and silica 98 % obtained in one hour, ash 12.88 % and silica 48 % obtained in two hour, ash 12.86 % and silica 72.2 % obtained in three hour, ash 13 % and silica 98 % obtained in four hour, ash 13.18 % and silica 98 % obtained in five hour, ash 10.94 % and silica 53 % obtained in six hour.

The amount of rice husk ash and silica obtained in 600°C is ash 24.94 % and silica 96.6 % obtained in one hour, ash 13.68 % and silica 62.8 % obtained in two hour, ash 12.72 % and silica 116 % obtained in three hour, ash 13.46 % and silica 65.4 % obtained in four hour, ash 13.2 % and silica 62.8 % obtained in five hour and ash 13 %, silica 68.4 % obtained in six hour.

The amount of rice husk ash and silica obtained in 700°C is ash 15.9% and silica 85.4% obtained in one hour, ash 13.02% and silica 51.2% obtained in two hour, ash 12.72% and silica 45.6% obtained in three hour, ash 13.26% and silica 77% obtained in four hour, ash 12.6% and silica 47% obtained in five hour and ash 12.88%, silica 34.4% obtained in six hour.

The amount of rice husk and silica obtained in 800°C is ash 13.08% and silica 60% obtained in one hour, ash 11.62% and silica 40% obtained in two hour, ash 11.72% and silica 20% obtained in three hour, ash 11.8% and silica 36% obtained in four hour, ash 11.24% and silica 20% obtained in five hour and ash 11.56%, silica 40% obtained in six hour.

Table 4.3: Extract silica from rice husk ash at different temperature

Tempratures(°C)	**Combustion time(hours) of rice husk**	**Ash%**	**H_2SO_4(5N)**	**NH_4OH**	**Silica %**
400	1h	50	45.5	4	46.4
	2h	46.6	45.3	3.6	33.4
	3h	38.2	45.4	3.5	50
	4h	32.6	45.5	3.1	48
	5h	32.2	40.5	3.85	40
	6h	32	40	3.8	42
500	1h	13.52	43.5	4.3	98
	2h	12.88	44.3	4.7	48
	3h	12.86	45.2	4	72.2
	4h	13	43.2	4.8	60
	5h	13.18	44.2	4.7	98
	6h	10.94	43.2	3.8	53
600	1h	24.94	45.2	4	96.6
	2h	13.68	44.3	3.5	62.8
	3h	12.72	43.7	4	96
	4h	13.46	43.2	4.3	65.4
	5h	13.2	43.6	4.3	98

Tempratures(°C)	Combustion time(hours) of rice husk	Ash%	H_2SO_4(5N)	NH_4OH	Silica %
	6h	13	43.5	3.45	68.4
700	1h	15.9	42.7	3.4	85.4
	2h	13.02	43.6	3.7	51.2
	3h	12.72	44.8	4.5	45.6
	4h	13.26	44.5	3.5	77
	5h	12.6	43.8	3.2	47
	6h	12.88	42.6	3.5	34.4
800	1h	13.08	44	3.8	60
	2h	11.62	44.2	3.5	40
	3h	11.72	44.7	4.3	20
	4h	11.8	45.7	4	36
	5h	11.24	45	3.5	20
	6h	11.56	45.6	3.7	40

The characterisation of the silica and activated carbon has been done by the scanning electron microscopy (SEM), Energydispersivespectrometer (EDS) and X-ray diffraction (XRD).

4.2.1 Scanning Electron microscopy (SEM)

TheSEM images of obtained silica at various temperature at 400°C, 500°C, 600°C, 700°C and 800°C and for processing time one hour and four hours are shown in Figure 4.6 & 4.7. The observation Figure 4.6 & 4.7 shows that the crystal of silica are successfully obtained for all the cases.

In Figure 4.6, the SEM images for silica crystal are shown for various temperature. The crystal of silica can easily seen for all cases. The crystal size are almost uniform in case of temperature 600°C for one hour opration time as shown in Figure 4.6.E. in other cases the particle size are non uniformand greater than 100 micrometer.

The Figure A, C, D and E shows that are the temperature of calcination increase from 400°C to 800°C. The particle size is increase little bit this statement is also confirm by the further of zoom

SEM images at shown the figure 4.6.I that regular silica particle structure with some fine particles. Where as for Figure 4.6.J the particle size distribution is little as than Figure 4.6.I and the particle size for other conditions are almost same so Hence we can say that the particle size above 500°C are almost regular. So the conditions above than 500°C is optimise for obtaining the silica particle. The SEM images also shows that silica particle size can be control by change in the temperature and calcination time.

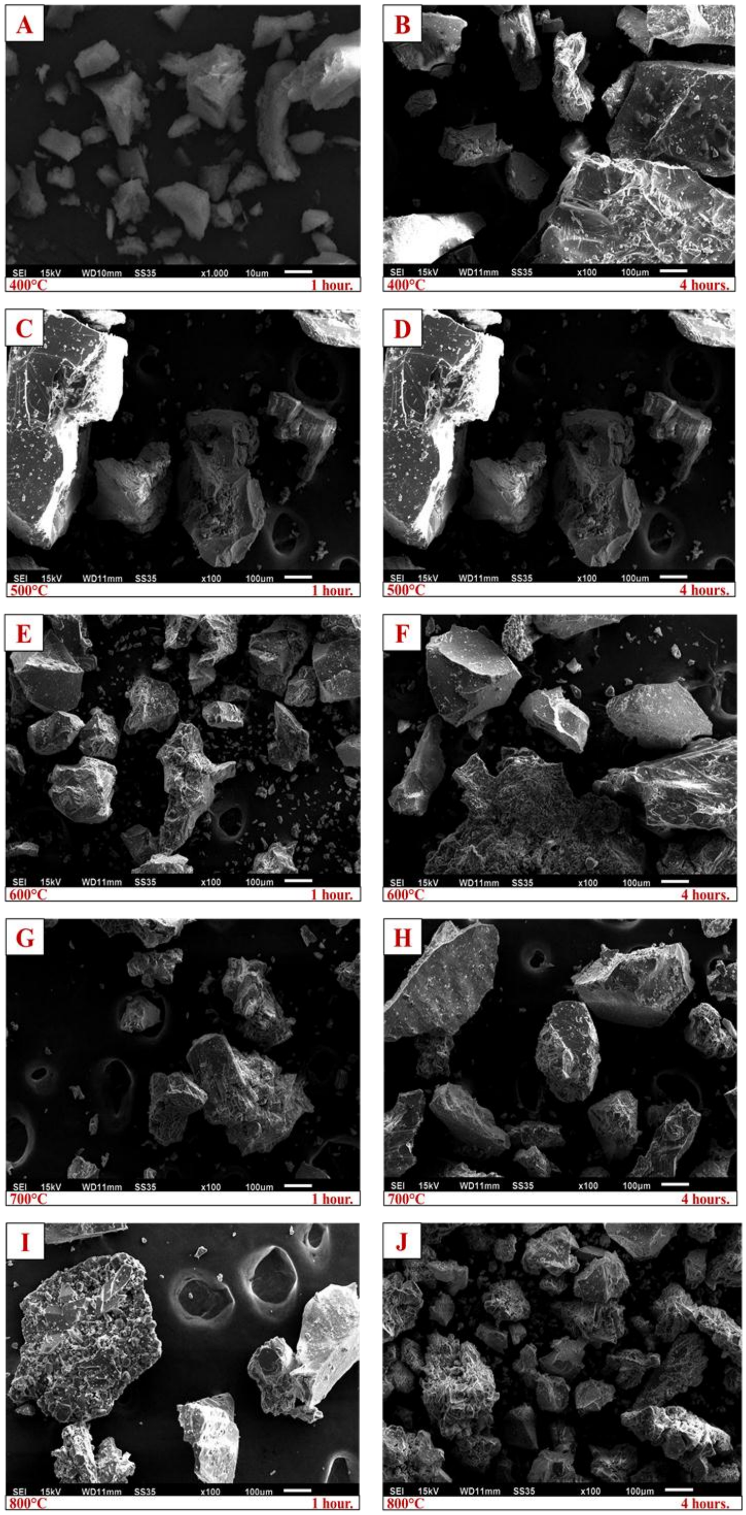

Figure 4.6: SEM images of silica (A) 400°C, 1hr (B) 400°C, 4h (C) 500°C, 1h (D) 500°C, 4h (E) 600°C, 1h (F) 600°C, 4h (G) 700°C, 1h (H) 700°C, 4h (I) 800°C, 1h (J) 800°C, 4h

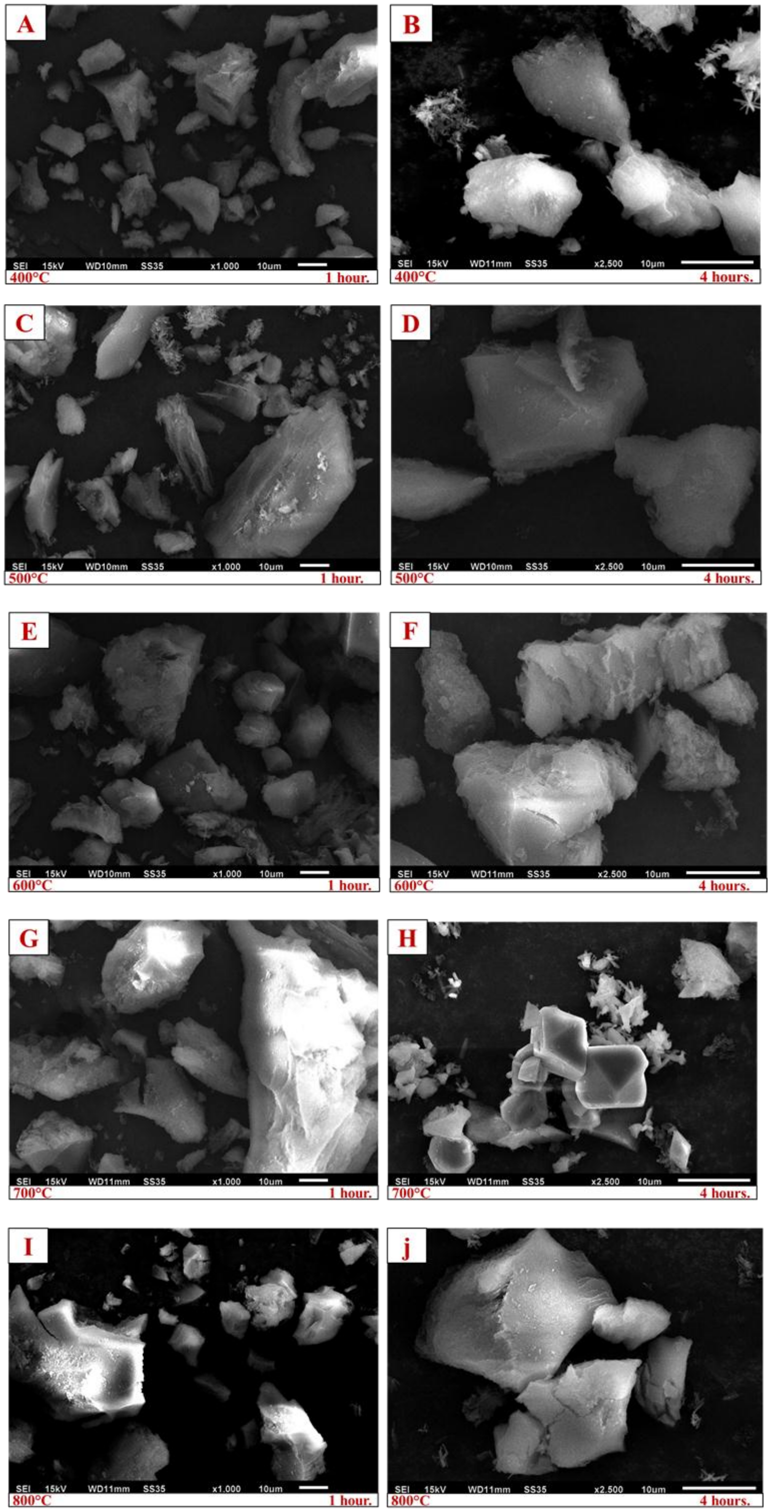

Figure 4.7: SEM images of silica (A) 400°C, 1h; x1000 (B) 400°C, 4h; x2500(C) 500°C, 1h; x1000 (D) 500°C, 4h; x2500 (E) 600°C, 1h; x1000 (F) 600°C, 4h; x2500 (G) 700°C, 1h; x1000 (H) 700°C, 4h; x2500 (I) 800°C, 1h; x1000 (J) 800°C, 4h; x2500

4.2.2 Energy-dispersive X-ray spectroscopy (EDS)

The Figure 4.8 shows that the 100% silica is obtained at all the temperature and processing time and the obtained resolution are Table 4.4. this table shows that there is no influence temperature for obtaining silica from rice husk but SEM images shows that silica obtained at 500°C having better and regular crystal structure.

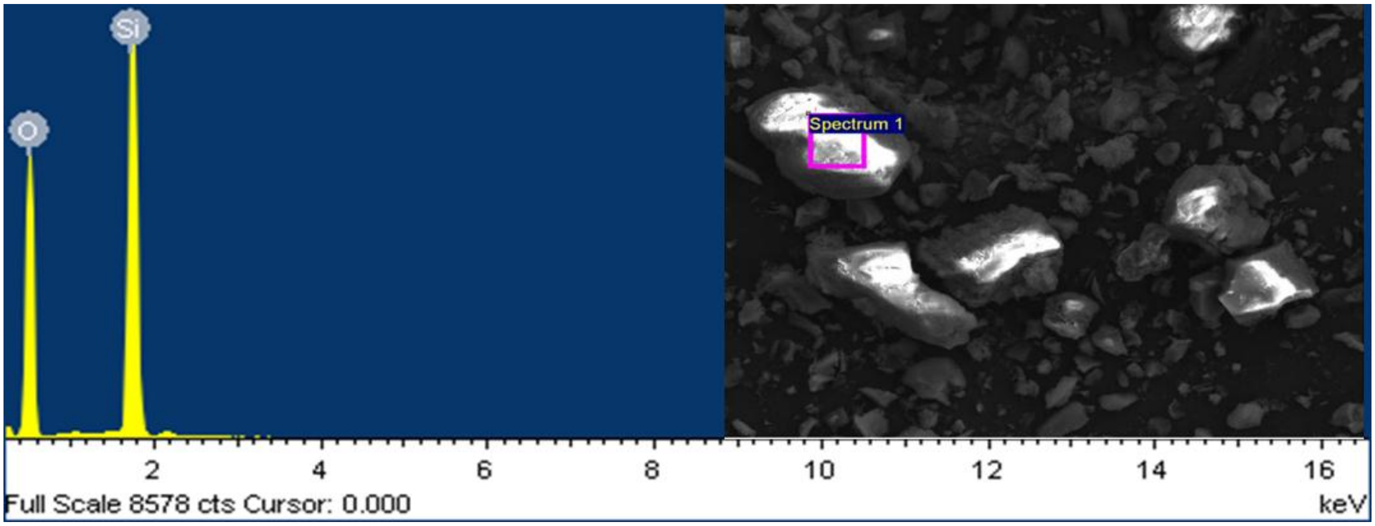

Figure 4.8:Energy-dispersive X-ray spectroscopy of silica (SiO_2)

Table 4.4: weight% of Silica obtained at different time and temperature.

S.No	Temperature (°C)	Time (h)	Silica (%)
1.	400	1	100
2.		4	100
3.	500	1	100
4.		4	100
5.	600	1	100
6.		4	100
7.	700	1	100
8.		4	100
9.	800	1	100
10.		4	100

4.2.3 X-ray diffraction (XRD)

XRD of obtained silica powder has been obtained and the results are summarized in Table 4.5. The observation table shows that the silicon is obtained up to 700°C with d-spacing in the range minimum 1.921 and maximum at 2 Theta 47.37 with crystallite size minimum 37 and maximum 53. The change in temperature resulted increase in crystallite size. At 800°C there are peaks for silicon as well silica. For silicon two peaks are obtained at 28.47, 47.35 with d- spacing 3.13184 and 1.91809 respectively.

The intensity of silicon at 28.47 is very high as compare to other theta values. For silica four peaks are obtained two theta of 42.398, 50.7148, 55.2316 and 68.1015 respectively.

The XRD confirmed that there is a mixture of silicon and silica at 800°C, whereas up to 700°C is able to obtained pure silica.

Table 4.5: Crystallite size and d-spacing of silica and silicon

S.No.	**Temp. (°C)**	**Sample**	**Crystallite size D_{XRD} (nm)**	**2 Theta**	**d- spacing (A°)**
1.	600	Si	37	47.27	1.921
2.	700		53	47.37	1.921
3.	800	Si	61	28.47	3.132
4.			60	47.35	1.918
5.		SiO_2	139	42.398	2.130
6.			61	50.75	1.799
7.			87	55.23	1.662
8.			39	68.16	1.376

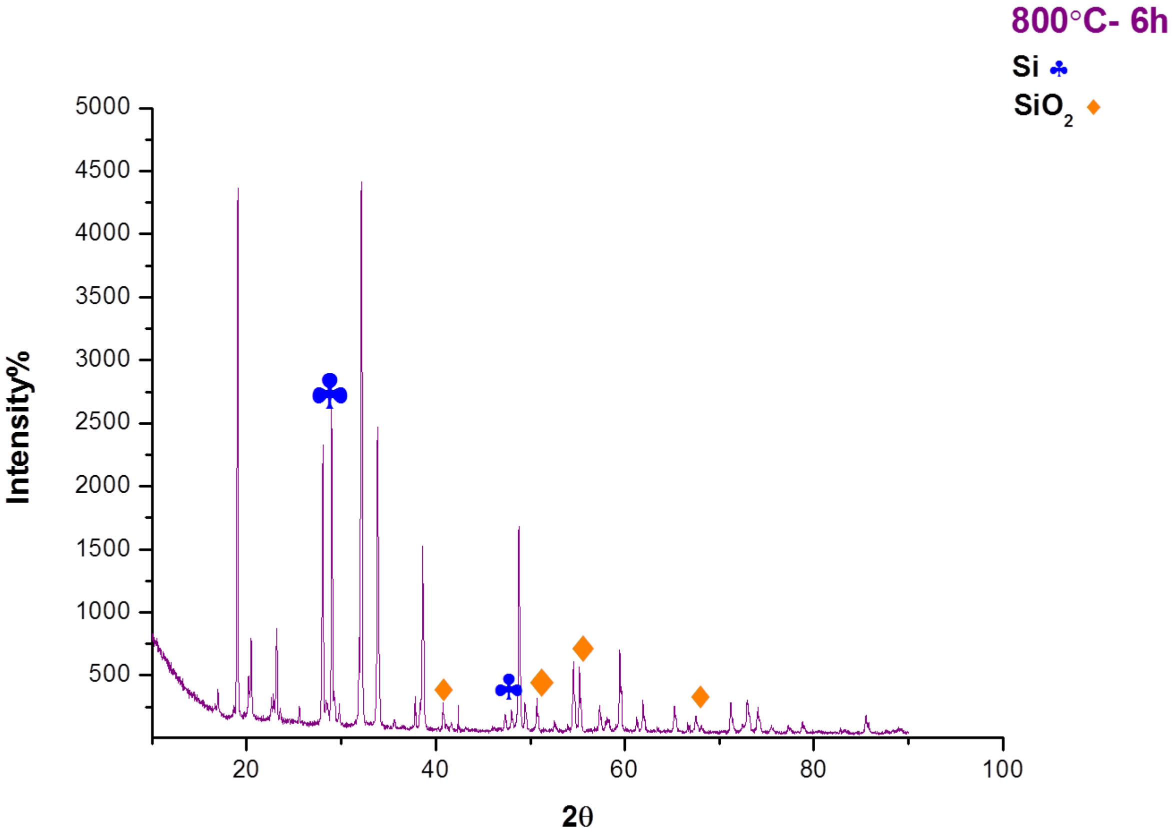

Figure 4.9: XRD pattern for nano silica at 800°C

4.2.4 Brunauer-Emmett-Teller (BET)

The BET of obtained silica has been performed to investigate the surface area and pore size. The result obtained BET is shown in the Table 4.6. The maximum BET surfaces has been obtained 280.68m^2/g at 500°C for six hours processing and minimum 27.52m^2/g at 600°C for six hours processing. The maximum pore volume obtained 0.655cm^3/g at500°C for six hours processing time and minimum pore volume 0.066cm^3/g at600°C for six hours processing.

Table 4.6: BET surface area of silica obtained at different time and temperature

S.No	Temp. (°C)	Time (hour)	BET specific surface area (m^2/g)	Total Pore volume (cm^3/g)	Average pore size (nm)
1	400	1	177.6	0.572	12.89
2		6	201.6	0.492	9.763
3	500	1	91.25	0.220	9.678
4		6	280.6	0.655	9.339
5	600	1	86.91	0.301	13.89
6		6	27.51	0.066	9.614
7	700	1	197.7	0.484	9.798
8		6	35.30	0.077	8.732
9	800	1	124.9	0.278	8.918
10		6	255.1	0.543	8.517

CONCLUSION

Rice husk ash has been obtained at various calcination temperatures and processing time.The activated carbon and silica has been extracting from rice husk ash.The activated carbon has been characterized using SEM, BET and EDS. The processing time of 14 hours is found optimum with larger pore size, regular structure and high pore density as per SEM images. The BET surface area, total pore volume and average pore size of the activated carbon are 4.683m^2/g, 8.886cm^3/g and 7.590nm respectively.

The maximum 74.63% weight and 72% weight of activated carbon were obtained for the observation time 14 hours and 12 hours respectively. The operating temperature was 100°C.

The silica is also extracted from rice husk at different calcination time and tempratures. According to SEM images the particle size at calcination temperature above 400°C are almost regular. The EDS Chracterization of silica shows that 100% silica is obtained at all the temperature and processing time. The XRD analysis of obtained silica is shown that the silicon is also present along with silica. Hence, The XRD analysis confirms that the obtained white powder is the mixture of silicon and silica. The BET surface area, total pore volume and average pore size of the silica are 280.68m^2/g, 0.6553cm^3/g and 9.339nm respectively.

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