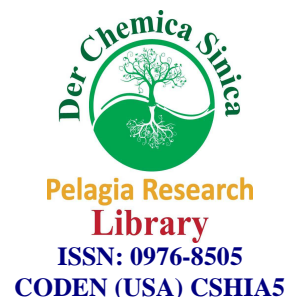




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Der Chemica Sinica, 2016, 7(2): 1-7



Utilization of silica extracted from soybean hulls for chromatographic studies of catechol amines

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ABSTRACT

Cultivation of soybean is extensively done in Malwa region of Madhya Pradesh nearly 80% of total soybean produced in India. And it is classified source of oil and protein. Its seed are dehulled and the hulls being by-product of these industries, therefore a preliminary attempt was made to utilize this waste for extraction of silica from it by sodium carbonate fusion followed by hydrolysis. The purity and percentage of extracted silica was estimated by conventional HF- method(hydro florid) and characterization is done by various physio-chemical techniques viz. IR, X-ray diffraction, TG pattern SEM, etc. The result shows absence of extraneous impurities in significant amount. The silica so obtained was used as adsorbent for chromatography study of Catechol amines to explore its practical utility. Good results were obtained.

Keywords – extraction, IR, X-ray diffraction, chromatography, TG pattern SEM etc

INTRODUCTION

The Soybean:-

It belongs to the family Leguminosae, sub family papilionoideae. It is an erect bushy annual plant of great morphological diversity. The mature pods contain one to four seeds each. It consists of seed coat surrounded by a layer and well developed embryo. The average chemical composition of seed is 40% protein, 20% lipid, 4% lignin, 3% ash. The seed coat makes up about 9% of the soybean by dry weight. It is composed of cellulose type material. There is very little oil in the seed coat. It contains about 9% protein¹.

Inspiration for work :-

In order to develop methods for waste utilization so as to overcome disposal problems². Efforts have been made by the institutions like RRL Jorhat, Central salt and maine chemical research institute Bhavnagar. IIT Kharagpur has developed poly crystalline silicon from rice husk³ a waste of rice milling Industry this information inspired and prompted to use soybean hull (a waste of soybean based Industry) for some useful purpose.

MATERIALS AND METHODS

The seed were dehulled and the hulls were subjected for ashing in a muffle furnace at about 1100°C taking known quantity of ash silica percentage is estimated by conventional method^{4A-4B}. It comes to be 7-8% now after estimation bulk extraction of silica was done for studying their physico-chemical characteristics for practical utility as adsorbent purpose.

1. Physicochemical Methods

1.1 Infrared spectroscopy studies⁵ :-

The IR spectra of extracted silica was taken and it was compared with commercially available silica gel. The spectra are almost identical.

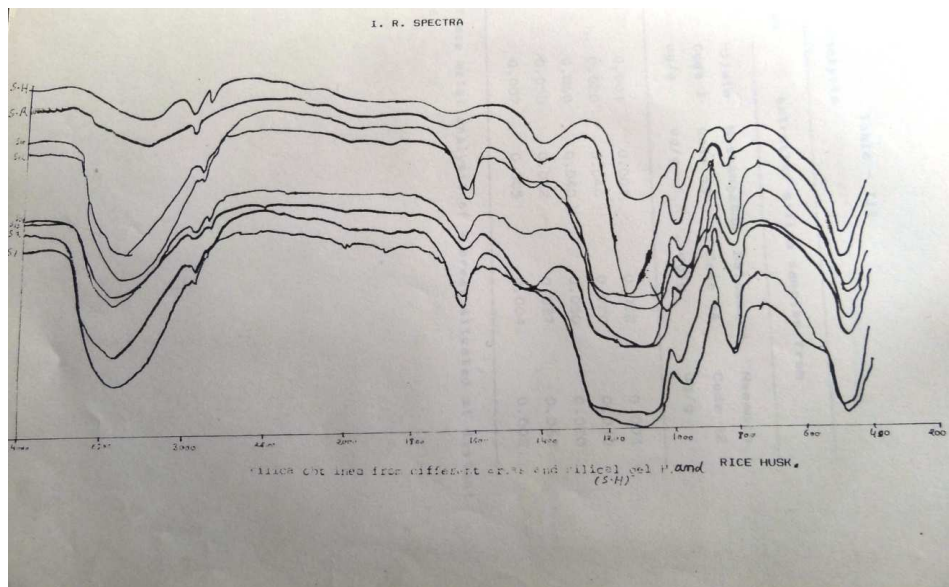
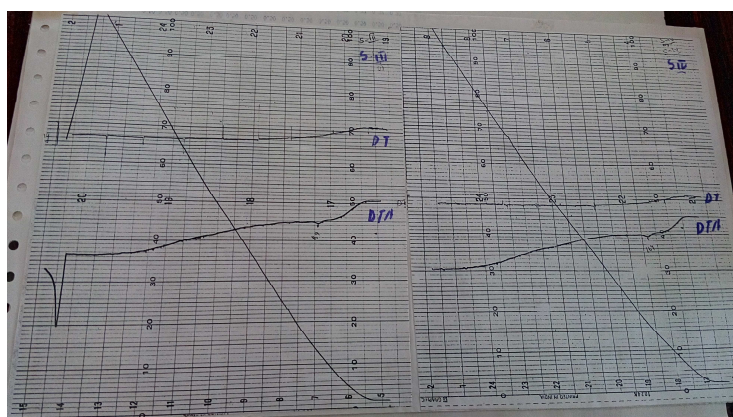


Table 1 I.R. frequencies cm^{-1}

Allocation	I.R. freq of S.G.H	I.R. Freq. of ext. Silica sample
OH stretching vib.	3500-3400	3500-3400
SiH stretching vib.	2920	2920
SiH stretching vib.	2840	2840
H ₂ O Bending vib. (deformation)	1600-1650	1600-1650
SiO Nati symm. Vib.	1050-1150	1050-1150
SiO Bending vib. (deformation)	960	962
SiO Anti Sym vib	800	800
SiO Bending vib. (deformation)	460-480	460-480

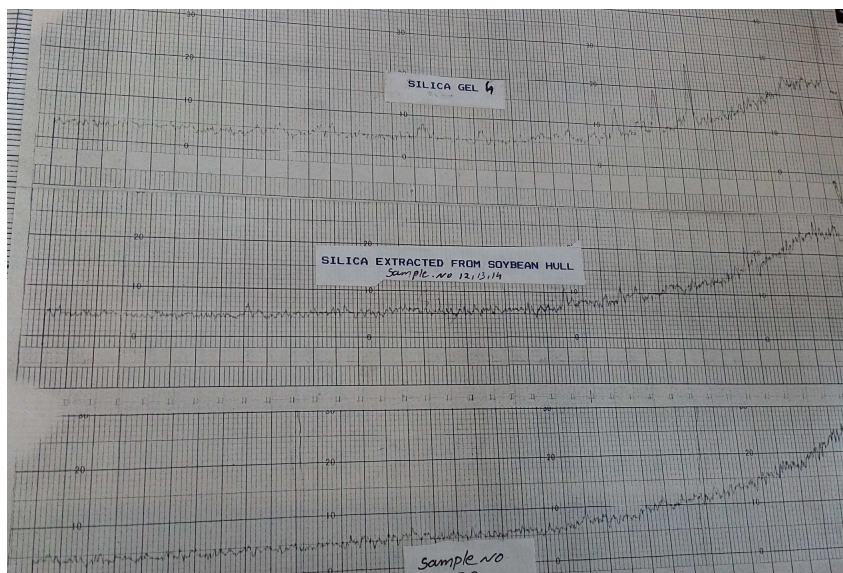
1.2 Differential Thermal DTA and Thermo-Gravimetric Analysis TG :-

The theoretical foundations of thermo gravimetric and DTA have been described by lehmann⁶. The weight and energy changes can be identified and measured very clearly. The TG and DTA studies of sample have been shown similar peaks are due to physically absorbed water no major weight loss has taken place.



1.3 X-Ray Diffraction Analysis :-

XRD patterns are used to analyse and determine the nature of the crystallinity, composition and also recognition of unknown solid or mineral phase. X-Ray analysis of extracted silica sample obtained the 2θ , d Spacings of the profile (heated at high temp.). Were calculated and the phase present in the sample were ascertained. Only one single peak major peak matches with cristobalite (d Spacings 4.04) this observation lead to a conclusion that extraneous imparities are absent⁷.



1.4 Trace metal Studies :-

Extracted silica samples were analyzed atomic absorption analysis. The results indicate that extracted silica sample do not contain extraneous imparities in significant amount.

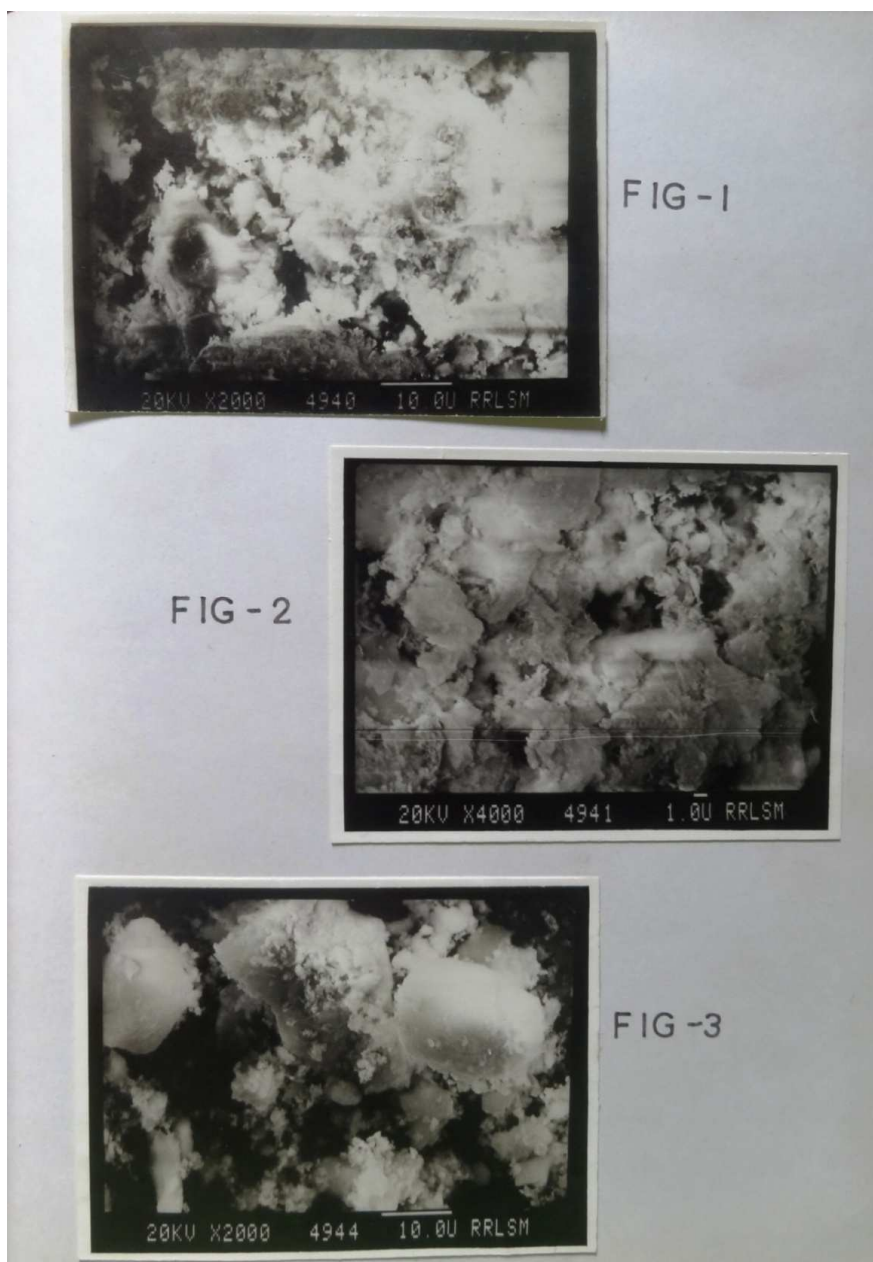
Table 2 Atomic Absorption Analysis :- Trace metal analysis

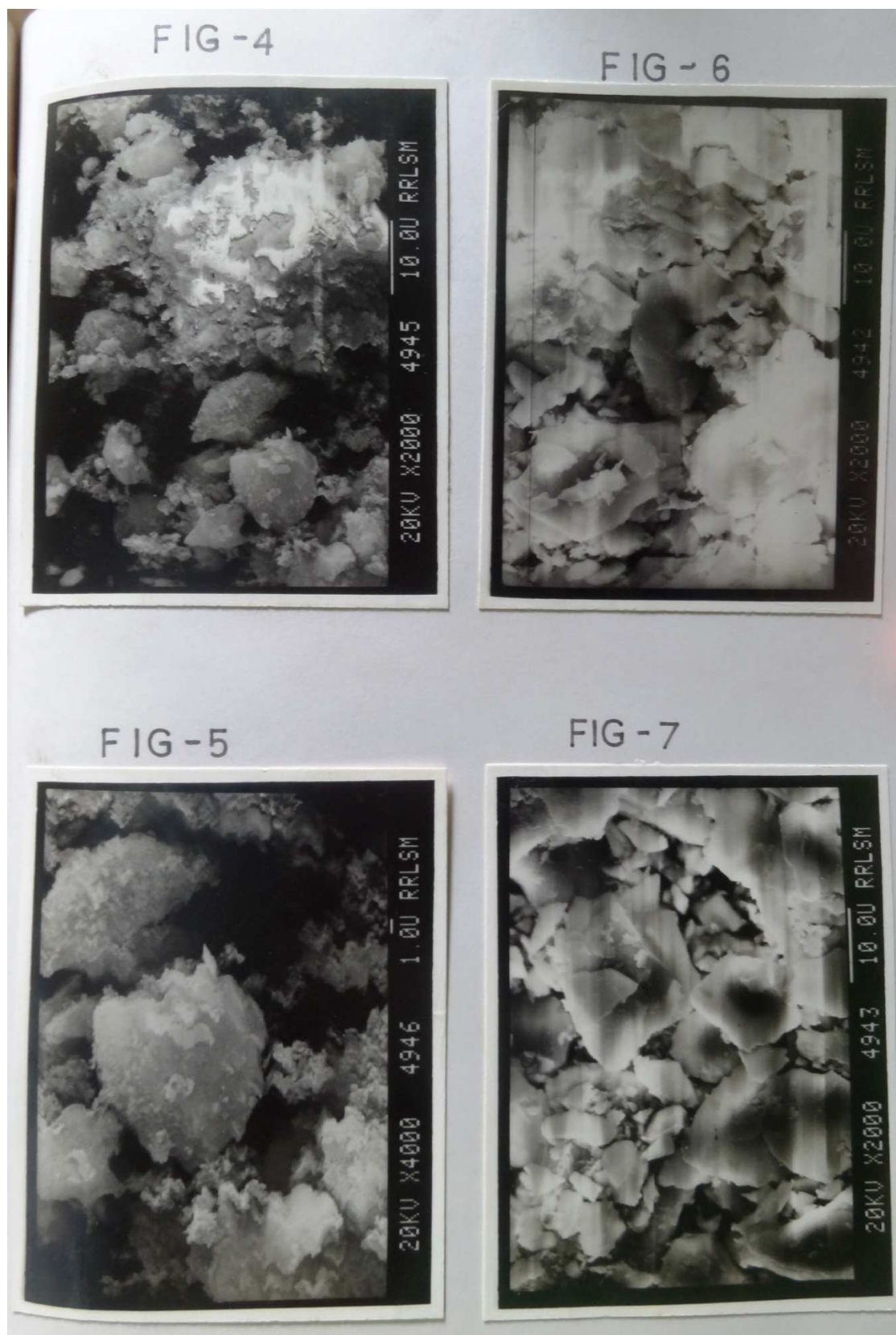
Name of imparities	Amount ug/g
Cobalt	0.001
Manganese	0.020
Lead	0.060
Copper	0.050
Nickel	0.007

1.5 Scanning electron microscopic studies :-

To compare the morphological characters using high magnification of the order of 2000-4000 times. The extracted silica and commercially available silica were examined these studies were carried out at RRL, Trivandrum and the results were photographed. The photograph indicate difference of crystallinity. The commercially available silica gel H exhibit coarse grind and certain degree of crystalline from where as extracted silica is not exhibiting crystalline form.

Fig of Scanning electron Microscope





1.6 Standardization and activity grades of adsorbent :-

Negligence of this factor has led many difficulties, contradiction and confusion. For standardization Brockman and shudder⁸ method is used and accordingly extracted silica has show activity grade II which is a class of activity useful for TLC.

1.7 Detection of an Impurity in extracted silica through TLC :-

The impurity either cationic, organic or inorganic nature or both type when introduced unknowingly in the adsorbent during manufacture or from the atmosphere they produce hindrance while performing experiment⁹⁻¹⁰ there for purity of extracted silica was also tested. The test shows absence of any extraneous impurity which will produce hindrance.

1.8 Having established that this novel adsorbent is of chromatographic grade chromatographic studies of separation of Cathecol amines were done.

The Cathecol amines are biogenic the mammalian adrenal gland is a compound gland consisting of an outer cortex and inner medulla. The hormones of medulla are amines derivative. Correctively referred as Cathecol amines. In which Cathecol as the basic nucleolus and amine attached to it. They can be separated on a silica gel layers. Using N-Butanol , Acetic acid and Water (8:2:10). They play an important role in the body.

(A) 0.1% Spot solutions of 6 Cathecol amines were prepared and to it very small amount of conc. Sulfuric acid was added in order to make them properly soluble. Spotting solution for binary mixtures separations were prepared by taking component by equal volume that is 1:1 that is volume by volume.

(B) Locating reagent – Iodine chamber the yellow spot on brown background were clearly detected.

(C) Solvent system used – n-Butanol-glacial acetic acid and water (8:2:10) (W/V) was used.

(D) Adsorbent for TLC :- The spot solutions of Cathecol amines from single component and binary component were spotted on to the chromate plates prepared by extracted silica and commercially available silica. The plates developed by conventional ascending technique in the development jar.

The investigations has revealed that the separation of the Cathecol amines taken in experiment is possible on extracted silica and is comparable to that on commercially available silica gel. The result shows resemblance in values obtained above.

The extracted silica shows potential applicability's to be used as TLC sorbent for Cathecol amines separation and study just like commercial sorbent.

Table 3

S.No.	Name of Amino Acid
1	3-4 dihydroxy phenyl alanine (DOPA)
2	3-4 dihydroxy phenyl ethylanin (DOPAMINE)
3	Serotonin
4	Adrenaline
5	Noradrenaline
6	Triocene

Table 4 TLC study of Cathecol amines with developed as individual spot

S.No. and code of Cathecol amines	RF on silica gel H layer	RF on extracted silica gel layer
1	0.23	0.21
2	0.58	0.58
3	0.65	0.65
4	0.38	0.37
5	0.50	0.51
6	0.00	0.00

Table 5 TLC Studies of Cathecol amines when developed as Binary mixture A & B

Binary mixture AB of Cathecol amines with code		RF on Silica gel H layer		RF on expctected silica	
A	B	A	B	A	B
1	6	0.23	0.00	0.21	0.00
2	6	0.58	0.00	0.58	0.00
3	6	0.65	0.00	0.65	0.00
4	6	0.39	0.00	0.37	0.00
5	6	0.50	0.00	0.51	0.00
1	2	0.23	0.58	0.21	0.58
1	3	0.23	0.65	0.21	0.65
1	5	0.23	0.50	0.21	0.51
3	4	0.65	0.39	0.65	0.37

RESULTS AND DISCUSSION

keeping in mind extensive investigation carried over on “Rice Husk” extracted silica. A detailed estimation study regarding presence of silica, percentage availability of it in hull ash were done and encouraged by result extraction of silica from hulls be done for physico-chemical investigation i.e., I.R., T.G., DTA, X-Ray, A.A.A., SEN, for characterization purpose.

All these investigations showed absence of any abnormal behavior and absence of any type of contamination in significant amount which will create hindrance for being used as an adsorbent. Because it is being biogenic and in plants purification process has already gone through in the nature when the organic material is absorbed from the soil in plants.

Considering all above facts a practical utility as an adsorbent for TLC has been explored by studying chromatographic behavior of Cathecol amines and compared with that of commercially available adsorbent. The data obtained resembles on both adsorbents. It has been concluded as good and novel adsorbent and hoped that it may find it's due place in the list of TLC adsorbents.

Acknowledgement

I feel greatly indebted to my Institute “Shri Vaishnav Institute of Technology and Science”. Who has inspired me to take this opportunity? I am thankful to Dr. Ing. V. P. Singh Director and Dr. V. N. Walivadekar Principal of my Institute for guidance and encouragement.

It is my sincere pleasure to thank all my colleagues specially Dr. Shishir Jain In charge of research development cell.

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