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Activated Carbon Produced from a Suitable Industrial Sludge

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ABSTRACT*:* The purpose of this study is the preparation of activated carbon using a suitable industrial sludge. The characterization of two types of industrial sludges showed that paper sludge was a better option over rice husk. The activated carbon produced from pyrolysis of paper mill sludge was chemically activated with various activating agents like zinc chloride, potassium hydroxide and potassium chloride. A systematic investigation of the effect of impregnation ratio, activation temperature and activation time on the properties of the activated carbon was done. The chemically activated carbons were characterized by measuring iodine and yield percentage. The activated carbon prepared from paper mill sludge in this study had maximum iodine of 764.80 mg/g with $ZnCl_2$ as the activating agent. The FT-IR analysis depicts the presence of a variety of functional groups. The XRD analysis reveals that the produced activated carbon has low content of inorganic constituents compared with the precursor.

KEYWORDS: Activated carbon, Paper sludge, Impregnation ratio FT-IR analysis, XRD analysis

I. INTRODUCTION

Activated Carbon is an essential substance for many industrial activities. For instance, bleaching agent (in sugar factory) and for water filtration. Most of the Activated Carbon for industrial activities is being imported from other countries. However, there is no sufficient amount of production to satisfy the need in our country and the demand for Activated Carbon in the market is high. So, to satisfy the demand the Activated Carbon is being produced using solid waste Rice Husk. The purpose of this project is the preparation of Activated Carbon using a suitable rice husk. The Activated Carbon produced from Pyrolysis of rice husk was chemically activated with activating agent sodium hydroxide (NaOH). The chemically activated carbons were characterized by measuring yield percentage and bulk densities. The activated carbon produced from rice husk at different activating temperature of 650ºC, 700ºC and 800ºC exhibit a yield percentage of 48.2%, 47.65% and 45.95% respectively and corresponding bulk densities were 0.2 g/ml, 0.16 g/ml and 0.117 g/ml respectively. Proximate analysis also performed for precursor selection to choose the appropriate precursor. The quality of activated carbon is highly proportional to the dehydration rate of the sample and also on the process of removal of the volatile substances present in the precursor. According to proximate analysis, rice husk has a volatile matter of 68.06%, ash content 0.952%, fixed carbon content 20.988% and moisture content of 10%. This contributes to a total volatile content (easily escapable components) of about 68.06%. The proximate analysis of rice husk also reveals that the selected rice husk has good carbon content which is 20.988%. Therefore, proximate analysis served as an evidence for choosing rice husk as the precursor. Finally, a preliminary material and energy balance on pyrolysis or carbonization was performed. The significant feature of sludge-based activated carbon that makes it a unique and a particularly economical adsorbent is that it can be produced from waste materials such as paper mill sludge or even the rice husk disposed of as waste from rice mills. A current problem faced by pulp mills is the generation of an excessive amount of sludge during the paper making process and secondary treatment of wastewater. For environmental and ecological reasons, the innocuous disposal of these sludges has become immensely important. Optimization of the processes involved with the conversion of paper mill sludge or rice husk to activated carbon provides an innovative, environmentally safe, and economically feasible solution to the problem of sludge management at paper mill facilities. Sludge to carbon conversion processes can significantly reduce the sludge volume produced in the paper mill industry or the rice mills, eliminate the need for further treatment of sludge, reduce the cost of hauling and land filling the sludge, and reduce transportation costs.

The emphasis of this study is to optimize processes involved with the production of activated carbons with prescribed surface properties (micro- or meso porous structure) and specific end uses from paper mill sludge. The higher purity (when compared to bio-solids), negative cost, high rate of production, and strong carbonaceous structure of paper mill sludge and rice husk makes them both useful as a precursor for carbon production.

II. LITERATURE REVIEW

[1] History, Method of Production, Structure and Applications of Activated Carbon (2017)

The process of adsorption is one of the physical method for separation of dissolved pollutants from the effluent. Activated carbon is the good adsorbent that can be used in both liquid and gaseous phase adsorption also used as catalyst or catalyst support. Adsorbent materials are porous and adsorption takes place on the inside walls of pore particles. From last few years, use of activated carbon has increased greatly. This review article purpose is to give the knowledge of how activated carbon uses came from historic days and also then how it is improving day by day. With this it gives the idea of production of activated carbon by different methods. The methods include physical activation and chemical activation. Adsorption capacity of activated carbon mainly depends on the structure of activated carbon.

[2] Adsorption of Lead and Iron from Industrial Wastewater using Melon (Citrullus Colocynthis) Husk Activated Carbon (2020)

The application of activated carbon as an adsorbent in remediating heavy metals from industrial wastewater is increasingly being embraced. Agro wastes activated carbon adsorbents have proven to be more suitable and cost effective than the conventional wastewater treatment methods for the remediation. In this work, the shell of melon (melon husk) were converted into activated carbon and applied for the adsorption removal of Iron (Fe^{2+}) and Lead (Fe^{2+}) ions. The melon husk carbon were activated using NaoH, H_2SO_4 and $CO(NH_2)_2$ (Urea) reactive agents. After which the adsorbent were then characterized using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). An adsorption study was then conducted; the wastewater were treated with activated adsorbent at a dosage interval of 0.2g, between 0 and 1.0g with a reaction time of 0 to 100 minutes at 10 minutes intervals and at constant speed of 150 rpm. The characterization study indicates that the melon husk ask is suitable for use as an adsorbent and the adsorption study indicate that the removal rate of these heavy metals varies with changes in activation method, contact time and adsorbent dosage. On the effect of dosage, the Melon husk activated carbon (MHAC) removed Iron (Fe^{2+}) ions between 6.39 - 22.81% for all activation agents at optimum dosage of 0.8g. While, 0-100% of Lead (Pb^{4+}) ions were adsorbed at dosage of 0.8g for all activation agent except for H_2SO_4.On the effect of contact time, equilibrium for removal of Iron (Fe^{2+}) and Lead (Pb^{4+}) was reached at an average contact time of 70minutes and 20minutes respectively for all activation agent. It was thus concluded that the Melon Husk Activated Carbon (MHAC) was more effective for removal of Lead (Pb^{4+}) ions compare to removal of Iron (Fe^{2+}) ions.

[3] Study on Preparation of Activated Carbon from Sludge (2013)

Sludge, a carbon-rich material and favourable precursor of activated carbon, has stimulated new methods for the production of activated carbons for water gas treatment. Herein, various influence factors, concluding sludge resource, pyrolysis process and the type of activators, of the preparation of sludge-based activated carbon have been compiled. Moreover, the mechanism of chemical and physical activation was analyzed. Finally, Then the main research directions of preparation of activated carbons from pyrolytic sludge also were proposed. Sludge is a byproduct during the process of sewage treatment. It is difficult to handle because of its complex constituent components. Organics is an important component of sludge. In order to utilize carbon in sludge reasonably, researchers convert sludge into activated carbon with adsorption. Because the raw material of sludge-based activated carbon is cheap and easy to obtain, and its adsorption ability to some pollutants is better than that of commercial activated carbon, the application of sludge-based activated carbon prospect is appreciable. Preparation of activated carbon from sludge can also achieve the goal of waste utilization. In this paper, the preparation of activated carbon from sludge is reviewed, and the development prospect of activated carbon from sludge is pointed out. The main way to prepare activated carbon from sewage sludge is pyrolysis or calcination. The adsorption capacity of activated carbon is mainly affected by factors such as sludge source, pyrolysis process and activating agent.

[4] A review on activated carbon: process, application and prospects (2016)

Activated carbon (AC) is used in different states of applications after its discovery as a strong and reliable adsorbent. An overview on AC is presented together with revisiting the sources of AC generation; methods used to generate AC comprising of pyrolysis activation; physical activation; chemical activation and steam pyrolysis. The important factors affecting the AC production, the possible applications of AC and their future prospects are also discussed. AC is applied in water, wastewater and leachate treatments in many countries, especially to polish the color, remove the odor and some heavy metals.

It is cheap and available, and can be produced from agricultural waste materials, e.g. rice husk, palm oil shell and coconut shell. The AC's fine and porous structure and an extremely large particle surface area (>1000 m2 /g) results in making it possessed powerful adsorptive properties. Therefore, the adsorption process using AC is found to be a potentially viable method of removing pollutants from aqueous solutions.

[5] Review on Activated Carbon: Synthesis, Properties And Applications (2021)

Many researchers have reported that a number of control methods were used in waste water treatment. In this work, there are several types of agricultural wastes and fruits were employed to synthesis activated carbon via chemical activation and physical activation process. The obtained activated carbons indicated higher surface area and larger adsorption capacity. This paper describes experimental findings on uses of activated carbon in wastewater treatment, which produced under different conditions such as contact time, initial pollutant concentration, temperature, pH value, adsorbent dosage, particle size and agitation.

III. PROPOSED METHODLOGY

Precursor (raw material) selection for the production of activated carbon was obviously the first step of the project. Conventionally, activated carbon is produced from carbonaceous source material such as wood, peat, coal, and wastes of vegetable origin (e.g. nutshells, fruit stones). Today, one promising approach for the production of cheap and efficient activated carbon is the reuse of waste sludge, such as bio solids produced at municipal or industrial wastewater treatment facilities. The usage of waste sludge is especially important due to its mass production and resulting occupation of valuable landfill space.

The two types of industrial sludge available as choices for precursor were:

1. ∟ Rice husk
2. ∟ Paper sludge

To decide the superiority of a precursor over the other characterization of the precursor was done
using various methods like:

1. CHNS elemental analyser
2. Proximate analysis

IV. RESULTS AND DISCUSSION

FT-IR ANALYSIS

The FT-IR spectroscopic study of the produced carbon. The sample showed four major absorption bands at 2900-3500 cm-1, 1300-1750 cm-1, 1000-1250 cm-1 and 450-750 cm-1. A wide band with two maximum peaks can be noticed at 2930 and 3450 cm-1.

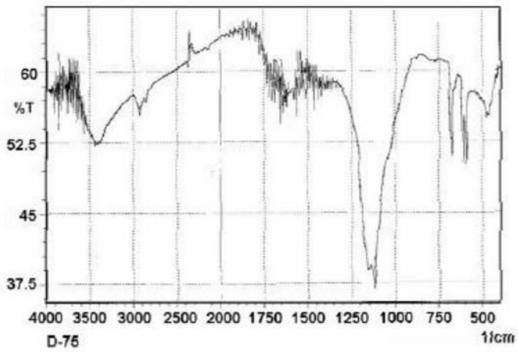
The band at 3450 cm- 1 is due to the absorption of water molecules as result of an O-H stretching mode of hydroxyl groups and adsorbed water, while the band at 2930 is attributed to C-H interaction with the surface of the carbon.

However, it must be indicated that the bands in the range of 3200-3650 cm-1 have also been attributed to the hydrogen-bonded OH group of alcohols and phenols. In the region 1300-1750 cm-1, amides can be distinguished on surface of the activated carbon which has two peaks at 1640 and 1450 cm-1. These functional groups were obtained during the activation process as a result of the presence of ammonia and primary amines that usually exist in the sludge. Moreover, the band at 1500 cm-1 may be attributed to the aromatic carbon–carbon stretching vibration.

The two peaks at 1125-1150 cm-1 yield the fingerprint of this carbon. The sharp absorption band at 1125 cm-1 is ascribed to either Si-O or C-O stretching in alcohol, ether or hydroxyl groups. The band at 1150 cm-1 can also be associated with ether C-O symmetric and asymmetric stretching vibration (-C-O-C- ring).

This band could also be attributed to the anti-symmetrical Si-OSi stretching mode as a result of existing alumina and silica containing minerals within the sludge samples.

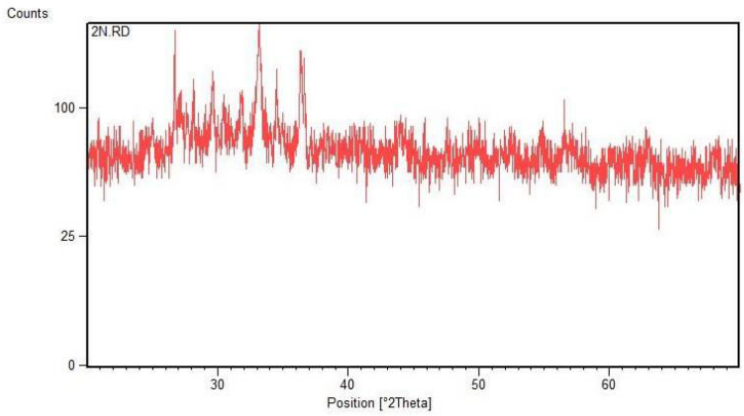
The region 450-750 cm-1 show two bands in the 480 and 485 cm-1 which are associated with the in plane and out-of-plane aromatic ring deformation vibrations. Peaks at 598 and 680 cm- 1 are assigned to the out-of-plane C-H bending mode. These spectra were also suggested to be due to alkaline groups of cyclic ketons and their derivatives added during activation.



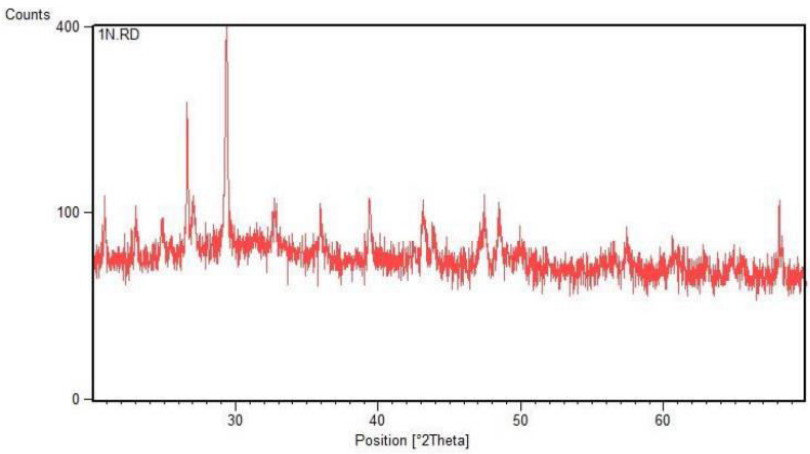
[Fig.5.1: FT-IR spectrum for activated carbon from sludge]

XRD ANALYSIS

X-ray diffractograms for both the activated sludge and activated carbon are shown in Figures 5.2 & 5.3. The XRD spectra of the activated sludge illustrated the presence of different aluminosilicate minerals. Zeolite X-Y was observed at 2θ = 29.4 with relative intensity of 158 cps, followed by faujasite detection at 2θ = 26.5o. Other peaks where located at 2θ = 32.9, 35.9 and 39.4o for mullite, hematite and quartz, respectively. While the rest of the peaks for sodalite, analcime and sodium silicates re located at 2θ = 43.1, 47.5 and 48.5.



[Fig.5.2: X-ray diffractogram for sludge sample]



[Fig.5.3: X-ray diffractogram for activated carbon from sludge]

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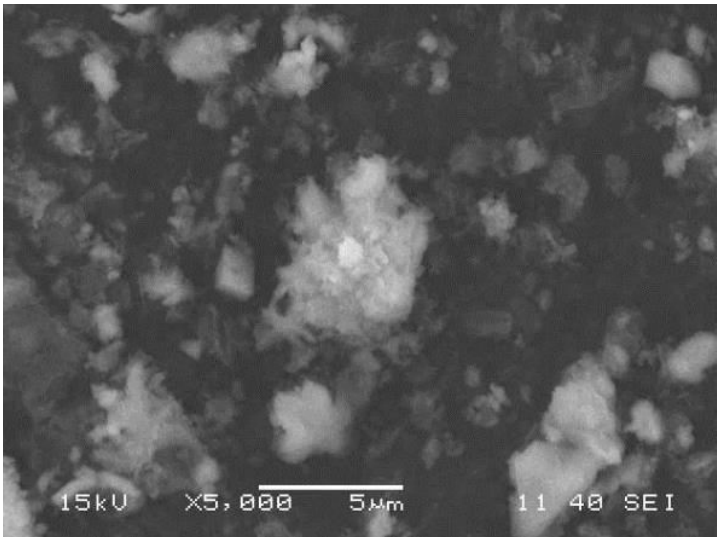
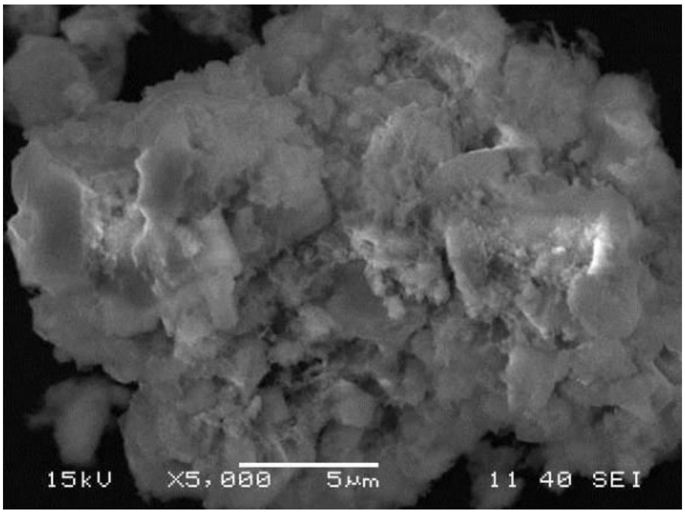
SCANNING ELECTRON MICROSCOPE ANALYSIS

The sample of activated carbon produced out of the best operating conditions like 2.0M $ZnCl_2$ activating solution, impregnation time of 20 h, activation temperature of 600 C for 1 h, was analyzed in a scanning electron microscope. The surface physical morphology of activated carbon was observed by a scanning electron microscopy (SEM) (S-2150, Hitachi High Technologies Corp., Japan).

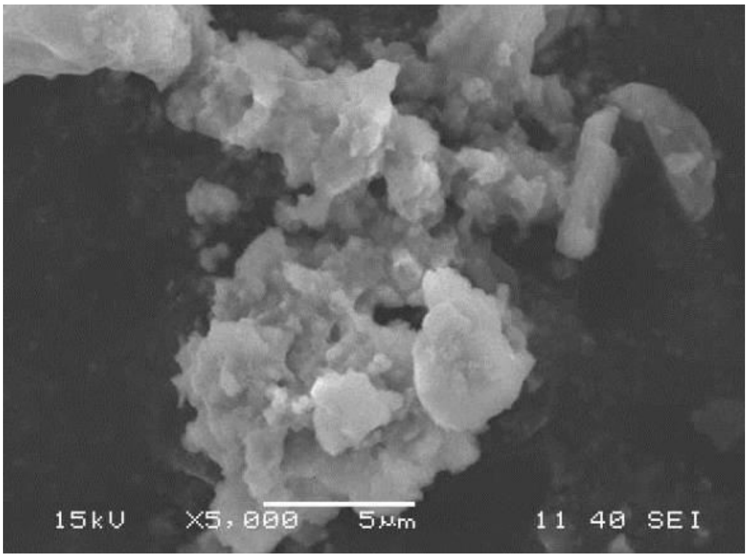
SEM photograph shows that wide variety of pores is present in activated carbon along with fibrous structure. $ZnCl_2$ and NaOH impregnated carbon consists of more canals like structure than the untreated carbon. In cases of $ZnCl_2$ and NaOH impregnated activated carbon, surfaces are pitted and fragmented. Those imperfections are not seen in case of untreated sludge.



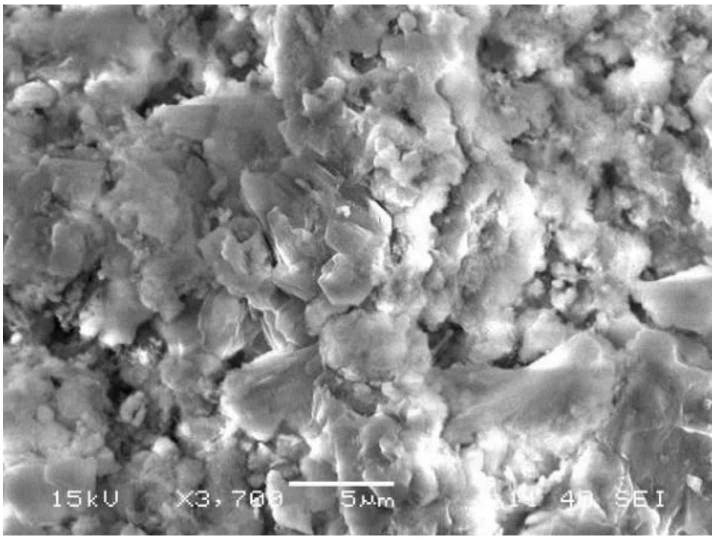
[Fig.5.4: SEM image of untreated sludge at 5000 times magnification]



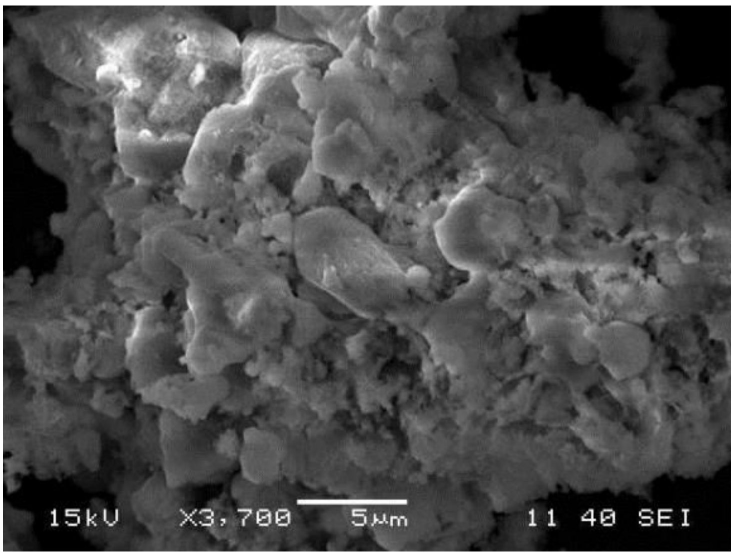
[Fig.5.5: SEM image of KOH activated carbon at 5000 times magnification]



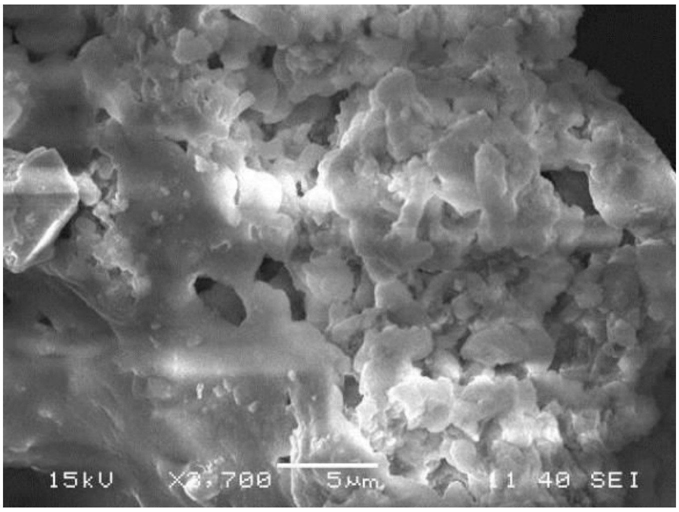
[Fig.5.6: SEM image of $ZnCl_2$ activated carbon at 5000 times magnification]



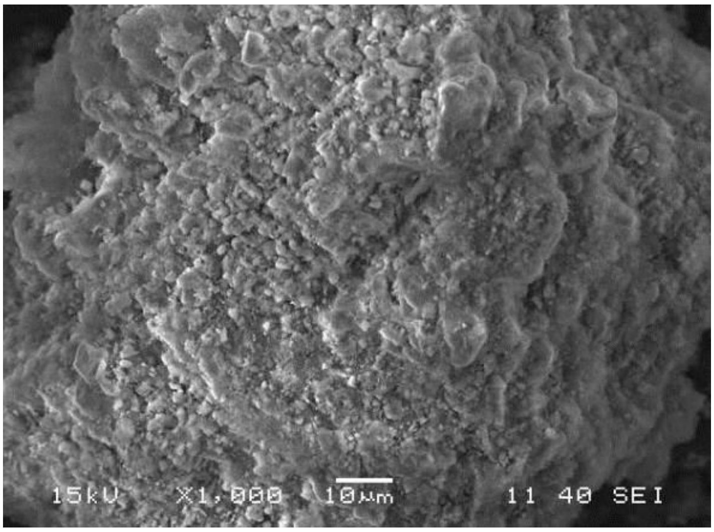
[Fig.5.7: SEM image of untreated sludge at 3700 times magnification]



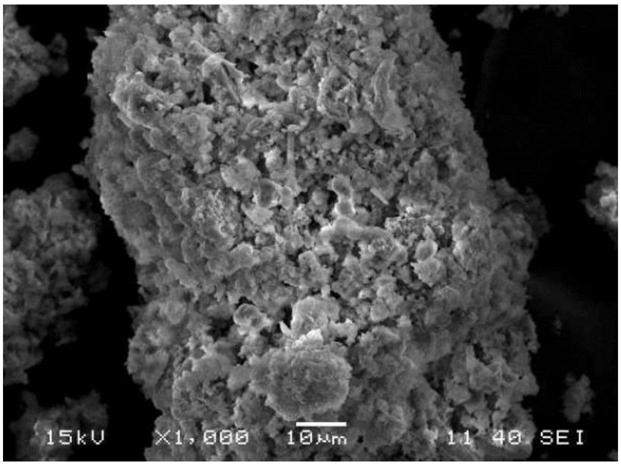
[Fig.5.8: SEM image of KOH treated activated carbon at 3700 times magnification]



[Fig.5.9: SEM image of $ZnCl_2$ activated carbon at 3700 times magnification]



[Fig.5.10: SEM image of untreated sludge at 1000 times magnification]



[Fig.5.11: SEM image of KOH treated sludge at 1000 times magnification]

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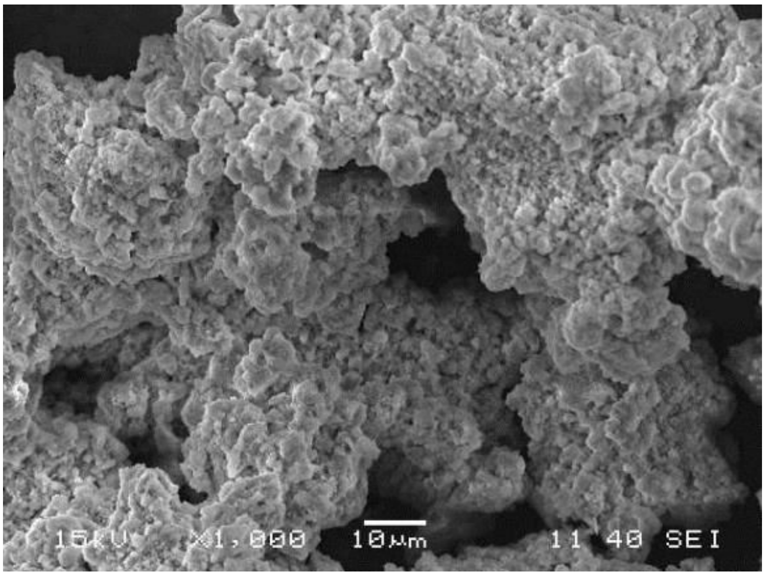

[Fig.5.12: SEM image of $ZnCl_2$ activated carbon at 1000 times magnification]

V.CONCLUSION

Activated carbon is a non-graphite form of carbon which could be produced from any carbonaceous material. Activated carbons are increasingly used as the economic and stable mass separation agent for the removal of surfactants to raise the final product quality many industrial processes. Activated carbons also play an important role in many areas of modern science and technology such as purification of liquids and gases, separation of mixtures and catalysis. Activated carbon are specially treated material which undergoes the chemical process to increase the adsorption capabilities of the material. Various material are used for the activated carbon which includes coal (anthracite , bituminous, sub-bituminous and lignite), coconut shells, wood(both soft and hard). Some materials have also been evaluate like wall- nut shells, olive stones and palm kernels. In our project we have used the coconut shells as the activated carbon material as there is a abundance of coconut farming in konkan area. The activated carbon using coconut shell will be economical in preface as the filter media with the slow sand filter at house hold level also. Activated carbon is a non-graphite form of carbon which could be produced from any carbonaceous material. Activated carbons are increasingly used as the economic and stable mass separation agent for the removal of surfactants to raise the final product quality many industrial processes. Activated carbons also play an important role in many areas of modern science and technology such as purification of liquids and gases, separation of mixtures and catalysis. The main objective of the study is to produce activated carbon from dry coconut shell and to treat the domestic waste water and to recycle the treated water for home gardens. The higher purity, negative cost, high rate of production and strong carbonaceous structure of industrial sludges proves to be a precursor for carbon production. This research will pave way for the recycle and reuse of waste water that could further reduce the level of water pollution.The results of this study show that it is feasible to prepare activated carbons with relatively high surface areas and pore volumes from paper sludge by direct chemical activation. An activation with $ZnCl_2$ produced activated carbons with better developed porosities than with KOH or KCl. The iodine value of the activated carbon product increased with concentration of $ZnCl_2$ solution (up to 2 N). As the impregnation time increased, the iodine value rose steeply, reaching a maximum value of 764.8mg/gm after 20 h. The iodine value also increased with activation temperature up to 600 C, beyond which it gradually decreased, presumably due to excessive carbonization. While prolonging the activation time, the iodine value of ZnCl2-activated carbon increased and then reached its maximum at 1 h. A longer activation time could induce negative effect on the carbon structure, and, thus, decrease the iodine value. In order to have a high surface area carbon and to minimize the energetic cost of the process, the following optimal conditions, 2.0M $ZnCl_2$ activating solution, impregnation time of 20 h, activation temperature of 600 C for 1 h were achieved. Under these conditions, activated carbon with a relatively high specific surface area of $737.6m^2$ /g and high iodine value of 764.8mg/ g was produced from paper sludge by direct chemical activation. The porosity of the product was comparable with that of commercially achieved carbon. The sludge-based activated carbon had a mean pore diameter of 6.72 nm, and its total pore volume and micro pore volume were 0.19 and 0.15cm3 /g, respectively, indicating its micro porous and meso porous character.

VI.FUTURE SCOPE

Nowadays, complicated challenges regarding environmental crisis because of pollution from various sources are faced all over the world, which eventually causes environmental degradation. Two decades ago, the environmental preservation measure to counter the aforementioned challenges has been one of the prioritized topics researchers are working on due to the large amount of agricultural waste production world over. The results found will immensely help in recovering contaminated resources like land and water bodies that surrounds our environment. Developing environment-friendly technologies after a careful study and targeting the status of green environmental policies, directed the interest of researchers in getting a supportive engineering measures and alternatives such as electro-sorption using AC. In practice, the adsorption capacity could directly be associated with the surface properties (i.e. pore size distribution, pore microstructure, surface area), accessibility, stability and permeability. In addition, the selection of appropriate electrodes plays an important role in electro-sorption assessment of systems particularly with regard to wastewater treatment. A simple pre-treatment (e.g. electrolysis) can be applied for wastewater and landfill leachate prior to activated carbon filtration to get high removal efficiency for various pollutants.

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