

Removal of Iron using Modified Low Cost Adsorbents

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Abstract –Water is one of the important renewable energy for all living and non living organisms. The concentration of the heavy metal like iron increases on water may cause bad effect to biotic life. To removing these heavy metals we use some method which is complex and uneconomical. Adsorption is one of the method widely used to remove heavy metal. We use some adsorbents like rubber seed shell, pistachio shell, tamarind pod shell, groundnut shell. The aim of this study was to produce a low-cost activated carbon derived from rubber-seed shells using potassium hydroxide (KOH) as chemical agent. The effect of preparation conditions such as impregnation ratio (1:1 and 1:2), temperature and duration 24 hours on the produced activated carbon were investigated. The results from adsorption test found that under the effect of initial concentration, the optimum sample is capable of removing iron. The obtained results show that agriculture waste product from rubber-seed shell is a promising low cost precursor for the production of activated carbon and it can be effectively used as an adsorption material.

Key Words: Adsorption, iron, pistachio, Tamarind pod shells, groundnut shells, Rubber seed shells, UV Visible Spectrophotometer

Date of Submission: 15-07-2022

Date of acceptance: 29-07-2022

I. INTRODUCTION

The uses of water is getting increased day by day. And also the essential of drinking water also increasing. But according to the study of World Water Council about billions of people didn't get enough drinking water. This is because of the water pollution. Now days water get more polluted that the people cannot take their enough water for their uses. The water get polluted by the industrialization, over exploitation of natural resources and population. Heavy metals refer to any metallic chemical element that has a relatively high density and very toxic or poisonous at low concentration with their accumulation over time can cause diseases like cancer to human while to environment, it is the cause of pollution. Heavy metal pollution derives from a number of sources, including, fuel and power manufacturing, agriculture industry, paper and pulp industry, leather tanning, electroplating, petrochemicals and fertilizers. Unlike organic contaminants, heavy metals such as Pb, Cd, Cr, Ni, Zn, Cu and Fe do not normally undergo biological decay and are thus considered a challenge for remediation. Accordingly, many studies have been done for removal of heavy metals. Chemical precipitation, reverse osmosis and ion exchange have been investigated for the removal process, but they are too expensive or incapable of meeting the acceptable levels before discharging to the environment. Adsorption technique by using activated carbon has been proved to be a potentially feasible alternative. However, the commercial activated carbon used is expensive to produce, particularly if proper raw materials are not available and the regeneration process involves high cost. Recently efforts have been made to use cheap and available agricultural wastes such as palm kernel, banana peel, coconut shell, doum seed coat and walnut shell as adsorbents to remove heavy metals from wastewater. Activated carbons (ACs) are predominantly amorphous solids with large internal surface areas and pore volumes. This unique pore structures plays an important role in many different liquid and gas phase applications because of their adsorptive properties. Surface morphology and pore structure of ACs depends on the precursors and selected activation agents. In this work, KOH is selected as the activating agent to produce AC from RSS due to excellent dehydrating properties and ability to develop and control the number of micropores with a very similar pore size distribution. On top of that, the preparation temperatures to activate the carbon were also relatively low, hence making the chemical activation process to be chosen over physical activation. The objectives of this study include the preparation, characterization and evaluation of adsorption properties of using RSS based AC to remove iron from single metal aqueous solution.

II.OBJECTIVE

- >To transform naturally available low cost seed shells to activated carbon.
- >To find the removal efficiency of adsorbents.
- >To modify adsorbents to increase efficiency.
- >To observe surface and porosity of modified activated carbon.
- >To validate the obtained results using minitab software.

III.IRON

Iron is a mineral that our bodies need for many functions. For example, iron is part of hemoglobin, a protein which carries oxygen from our lungs throughout our bodies. It helps our muscles store and use oxygen. Iron is also part of many other proteins and enzymes. Your body needs the right amount of iron. If you have too little iron, you may develop iron deficiency anemia. Causes of low iron levels include blood loss, poor diet, or an inability to absorb enough iron from foods. People at higher risk of having too little iron are young children and women who are pregnant or have periods. Too much iron can damage your body. Taking too many iron supplements can cause iron poisoning. Some people have an inherited disease called hemochromatosis. It causes too much iron to build up in the body.

IV.ADSORPTION

Adsorption has emerged as promising technique for metal removal. Among the various methods, adsorption is widely used for the toxic metal removal due to its higher removal percentage, simplicity and effectiveness. Adsorption can be described as a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid, forming a molecular or atomic film. The materials which is adsorbed is known as adsorbate and the surface to which it is adsorbed is known as adsorbent. Depending upon the forces acting between adsorbent and adsorbate. The adsorption process can be categorized into physical adsorption and chemisorption. Physical adsorption is the result of intermolecular forces of attraction between molecules of the solid adsorbent and the substance adsorbed. It is a readily reversible phenomenon. Chemisorption is the result of chemical interaction between the solid adsorbent and the adsorbed substance. Chemisorption process is irreversible and is mainly used in catalysis. Adsorption is operative in most natural, physical, biological and chemical systems and is widely used in industrial applications such as for preparing activated charcoal, synthetic resins and water purification.

V.MINITAB

Minitab is a software package that helps you to analyse data. Use Analyze Response Surface Design to model curvature in your data and identify factor settings that optimize the response. Usually, you use a response surface design after you have conducted a factorial or fractional factorial experiment and have identified the most important factors in your process. Before you can analyze your data, you must use Create Response Surface Design. Create Response Surface Design or Define Custom Response Surface Design to enter or define your design. You also need to have response data in the worksheet. For example, an engineer wants to analyze the injection molding process for a plastic part. After performing a fractional factorial design to identify the important factors (temperature, pressure, cooling rate), the engineer uses a response surface design to analyze curvature in the data and find the best factor settings. So we create a responses surface that by using central compositing which have included number of continuous factor. The factors are initial concentration, adsorbent dosage, pH, contact time. We just give these parameters low and high values on continuous factor. From this we just optimizing the value. The optimized values is used for further experiment. The values are initial concentration - 50ml, adsorbent dosage - 1gm, pH - 6, contact time - 150 min.

VI.EQUIPMENT USED

Table 1: list of equipment used

NAME	SPECIFICATION
Hot air oven	Working temperature 50 .C to 250.C
UV visible spectrophotometer	200-900 (±0.5nm)
Magnetic stirrer	Stirring speed 1200 RPM
Fourier transform infrared spectroscopy	
Minitab	

VII.METHODOLGY

A Preparation of adsorbent

The four adsorbents were collected. Rubber seed were collected from a rubber plantation, tamarind were collected from anicadu, pistachio and groundnut brought from amazon. The collected shells were washed using

distilled water and later dried in oven at a temperature of 200°C for 24 hours. The dried samples were taken out, crushed and sieved through Indian Standard sieves. The particles that passed through 300µm sieve and retained in 150µm sieve were collected and stored in an air tight container for further use.

B preparation of KOH solution

The experiment aims at removing iron from water using natural low cost adsorbents. For this the adsorbent were want to dipped freshly prepared KOH solution. This KOH solution where prepared in the laboratory by using KOH flakes which was purchased from a chemical shop in Thodupuzha. We need one normality KOH solution, so 56.11g KOH flakes were taken. This were added to 1 litre of distilled water and stirrer it well, thus we get 1 N KOH solution.

C Preparation of each adsorbents

20g of each adsorbent powder where measured and dip in 200ml KOH solution separately. These were done at room temperature for 24hrs. After 24hrs this each solution which the adsorbent powder dipped is filtered by filter paper. The sieved powder where kept first in sundry and this where placed at oven for dry. The oven dry is done at 80°C for 6hrs. After 6 hours the adsorbent powder taken and stored in beaker. Iron test is done on this powder.

D Preparation of synthetic iron

The experiment is to be done for the removal of iron from the water using natural low cost adsorbent. For the work synthetic industrial waste water was prepared in the laboratory by using some chemicals. For the this take 50ml distilled water and add 20ml concentrated H₂SO₄ drop by drop. Measure 1.404g of Fe(NH₄)₂(SO₄)₂·6H₂O add this to the above one. Add 0.1 N KMnO₄ drop by drop till the pink colour get constant. Dilute this with distilled water to get 1000ml of solution.

E Iron test on each adsorbent

Standard iron test is done to the one of the iron sample and find the concentration of iron in the solution by spectrophotometer. We prepared another four samples for the testing of four adsorbent. Dipped the four adsorbents to four sample and allowed to magnetic stirring for 10 minutes at 100 rpm. After 10 minutes stirring these four samples were allowed to rest for one and half hour and filter this samples by using A4 filter paper. Standard iron test is to be done for all these four samples and find the percentage adsorption for each adsorbents.

VIII. RESULT

The concentration of iron in first sample is 4.38mg/l. The concentration of iron for second sample which is pistachio shell powder dipped is 0.74647mg/l and thus the percentage adsorption is 82.95%. The concentration of iron for the third sample which is groundnut shell powder dipped is 0.88732mg/l and the percentage adsorption is 79.74%. The concentration of iron for the fourth sample is 1.0704mg/l and the percentage adsorption is 75.56%. The concentration of iron for the fifth sample is 1.2253mg/l and the percentage adsorption is 72.025%. So we get adsorption rate as pistachio then groundnut then tamarind then rubber.

IX. FTIR ANALYSIS

FTIR is the fourier transform infrared spectroscopy is a material analysis technique which helping identify organic and some inorganic material that could be the source of product of contamination or cause a malfunction. It having a principle that many gases absorb infrared radiation at its species-specific frequencies. By applying infrared radiation to sample, FTIR analysis measure a sample adsorbance of infrared light at various wavelength. This determine the materials molecular composition and structure, FTIR can be used for solids, liquids, and gaseous sample. Molecular bonds vibrates at various frequencies its depending on the element and the type of bonds. These where identify the functional group and molecular structure in the adsorbents. We want to known the functional group and molecular structure of four adsorbents. So that we go for the FTIR test on each of four adsorbents.

A Analysis of pistachio shell powder

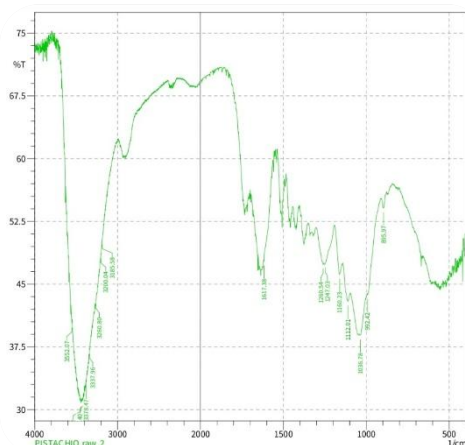


Fig 1: pistachio shell raw

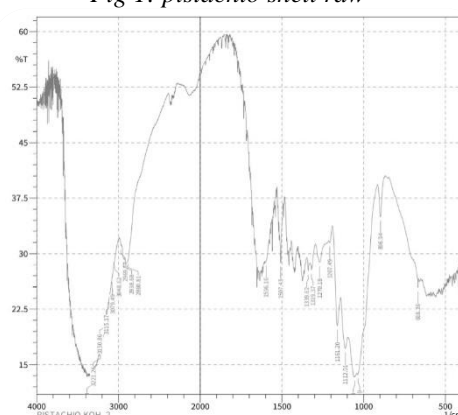


Fig 2 : pistachio KOH

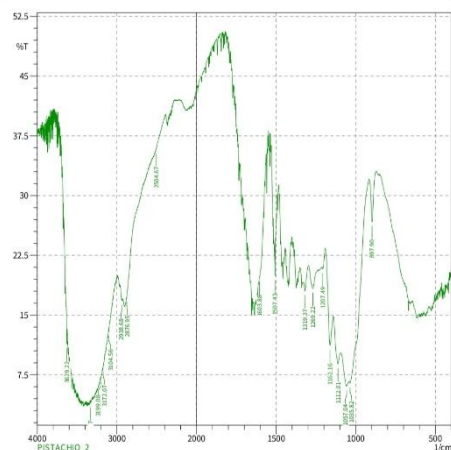


Fig 3 : pistachio

Fourier Transform infrared spectroscopy analyses were done to identify the functional group and molecules structure present in the adsorbent. The spectrum range of FTIR was within 4000 to 500 cm^{-1} . FTIR of pistachio raw powder (fig 1) pistachio dip in KOH (fig 2) pistachio after experiment (fig 3). We have more than 5 peak so we can see it's a complex compound. In pistachio raw strong peak at 3552.07cm^{-1} . There is no broad adsorption band in range between 3650 to 3250cm^{-1} that indicate the absence of hydrogen bond. There is peak in this area. It shows the sharp intensity of adsorption it allows the compound to contain an oxygen related group, such as alcohol or phenol we can see narrow band above 3000cm^{-1} that indicate unsaturated compound or aromatic ring. Narrow band below 3000cm^{-1} shows. It shows is the absence of aldehyde. We didn't find any peak between 2000 - 2500cm^{-1} so we can concluded that there is no triple bond region. Inform the absence of carbon triple bond in material. To identify the double bond region, we need to check between 1500 - 2000cm^{-1} . We have small intensity bands or small peaks above 1775cm^{-1} informing active carbonyl groups such as anhydrides halide acids etc. In range between 1750 - 1700 . We have narrow picks. Its indicate carbonyl

compounds such as ketones, ester etc. below 1700cm^{-1} implying amides or carboxylate functional groups. In peak in $1670\text{-}1620\text{cm}^{-1}$ shows the presence of unsaturation bonds. Specially we have a peak at 1650cm^{-1} . It is for double bond carbon or olefin compound. In 1617cm^{-1} we have a sharp peak that informs about aromatic compound. The peaks between $1500\text{-}600\text{cm}^{-1}$ show the fingerprint region.

In FTIR the powder shell of pistachio that dipped in KOH shows high peaks at 3334.1cm^{-1} as for pistachio powder after experiment shows high peak at 3629.22cm^{-1} . The chemical compounds present in these powder remain the same. But there is a change in intensity of absorption causing a change in peaks. It may be caused by KOH and also due to the adsorption process.

In raw pistachio c=shell powder at peak in 895.97cm^{-1} shows an area of 6.064. In pistachio shell powder dipped in KOH at peak in 896.94 shows an area of 16.34 per unit. And for pistachio shell powder after experiments at peak in 897.9 has an area of 20.193 per unit. It shows an increase in area.

Analysis of ground nut shell powder

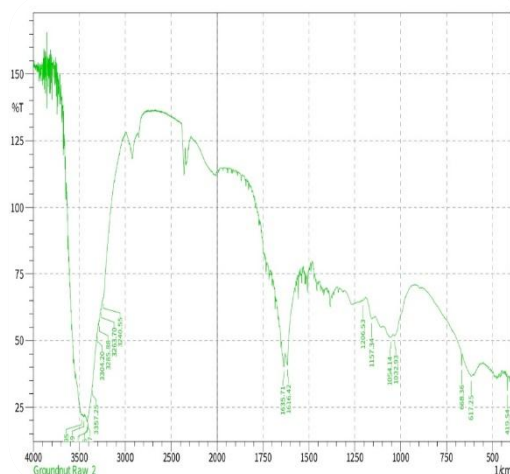


Fig 4 : groundnut raw

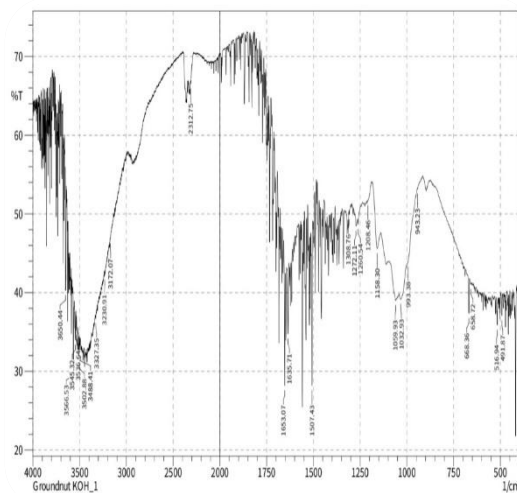


Fig 5 : groundnut KOH

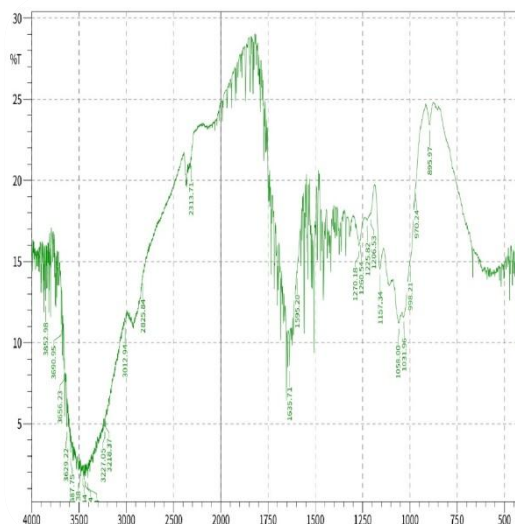


Fig 6 : groundnut

FTIR graph analysis for raw groundnut shell powder says that the spectrum has more than 5 adsorption bands, the sample can be complex molecules. The downward spike is maximum at 22% transmittance where adsorption band is at the range between 3000 to 3500 cm^{-1} . The stretching characteristics vibration band associated with the bonds in some common functional groups seen in this FTIR spectrum are at a range of 3000 to 3500 cm^{-1} a broad O-H bond stretching (alcohol) with 22% transmittance of light and at a range of 1650 to 1750 cm^{-1} a strong C=O stretching (Carbonyl) bond is seen with above 40% of transmittance of light and a fingerprint region of 1250 to 1000 cm^{-1} above 45% transmittance of light occurs.

FTIR graph analysis for groundnut shell powder dipped in KOH says that the spectrum has more than 5 adsorption bands, the sample can be a complex molecule. The downward spikes at below 35% transmittance where adsorption band is at a range of 3650.4 cm^{-1} wave number. The characteristics stretching vibration bands associated with the bonds in some common functional groups seen in this FTIR spectrum are at 3650 cm^{-1} a broad OH bond stretching (alcohol) with below 35% of transmittance of light and at a range between 1750 to 1500 cm^{-1} the position of C=O stretch varies slightly by carbonyl functional groups. This ranges include aldehydes, ketones, esters etc... which shows so many peaks in that region at fingerprint region transmittance of light is above 40%.

FTIR graph analysis for groundnut shell powder after experiments says that the spectrum was more the 5 adsorption bands, the sample can be for a complex molecule. The downward spike is maximum at below 5% transmittance where adsorption band is at a range of 3852.98 cm^{-1} the characteristic stretching vibration bands associated with the bond is some common functional groups seen in this FTIR spectrum are at 3852.98 cm^{-1} a broad O-H bond stretching (alcohol) with below 5% transmittance hence the peak is a wider peak and so many small peaks are seen throughout hence C=O bond and transmittance above 10% is seen.

Raw groundnut shell powder shows a surface area of adsorption 21.11 per unit area for an adsorption band of 1032.93 cm^{-1} . Groundnut shell powder dipped in KOH solution shows a surface area of adsorption 17.17 per unit area for an adsorption band of 1032.93 and groundnut shell powder after all experiment show an adsorption band of 1031.96 at 28.938 per unit area. This indicate that groundnut shell modified with KOH and powder obtained after experiments became more able to make an adsorption of light in a small surface area with the help of strongest O-H bond available and hence a lot of area is remaining for adsorption in the case of groundnut shell powder mixed with KOH. But so many adjacent number of downward spikes shows number of bonds acting as the reason for adsorption therefore the adsorption increase with increase in surface area relation cannot be found out without comparing the number of peaks.

C Analysis of tamarind pod shell powder

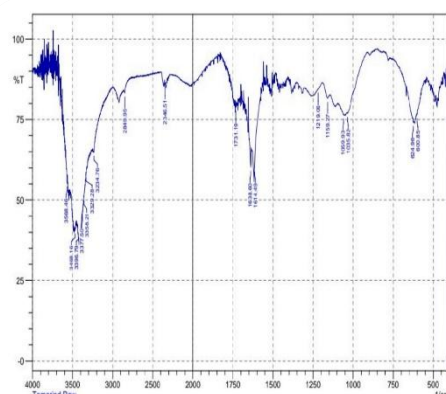


Fig 7: tamarind pod raw

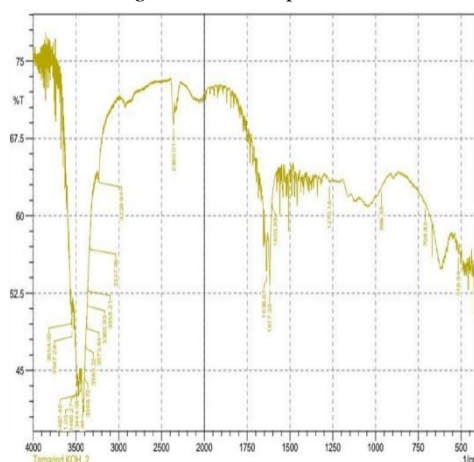


Fig 8 : tamarind pod KOH

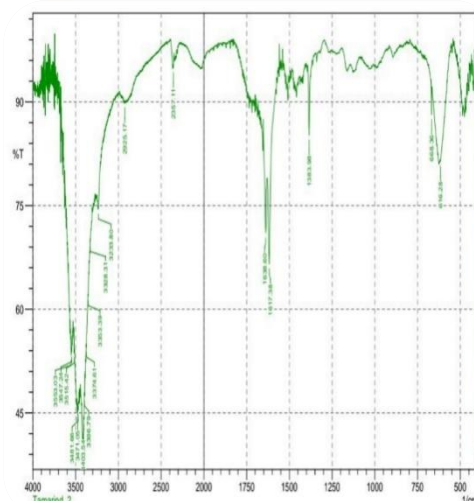


Fig 9: tamarind pod

FTIR graph analysis for tamarind pod shell powder (raw) says that the spectrum has more than 5 adsorption bands, the sample can be a complex molecule. The downward spike is maximum at 35% transmittance where adsorption band is at range of 3500 cm^{-1} wave number. The characteristic stretching vibration bands associated with the bands in some common functional groups seen in this FTIR spectrum are at 3500 cm^{-1} a broad O-H bond stretching (alcohol) with 35% of transmittance of light and at 1650 cm^{-1} a strong C=O bond stretching (carbonyl) with 60% of transmittance of light

FTIR graph analysis for tamarind pod shell powder dipped in KOH says that the spectrum has more than 5 adsorption bands, the sample can be a complex molecule. The downward spike is maximum at below 45% transmittance where adsorption band is at range of 3500 cm^{-1} wave number. The characteristic stretching vibration bands associated with the bonds in some common functional groups seen in this FTIR spectrum at

3500 cm^{-1} a broad O-H bond stretching (alcohol) with below 45% of transmittance of light at 1650 cm^{-1} a strong C=O bond stretching (carbonyl) with 52.5% of transmittance of light.

FTIR graph analysis for tamarind pod shell powder after experiment says that the spectrum has more than 5 adsorption bands, the sample can be a complex molecule. The downward spike is maximum at below 45% transmittance where adsorption band is at a range of 3400 cm^{-1} wavenumber. The characteristic stretching vibration bands associated with the bonds in some common functional groups seen in this. FTIR spectrum are at 3400 cm^{-1} a broad O-H bond stretching (alcohol) with below 45% of transmittance of light and 1650 cm^{-1} a strong C=O bond stretching (carbonyl) with 66% transmittance of light.

Raw tamarind pod shell powder shows a surface area of adsorption 3.9416 per unit area for an adsorption band 3396.79 cm^{-1} . Tamarind pod shell powder dipped in KOH shows a surface area of adsorption 2.918 per unit area for an adsorption band of 3398.72 cm^{-1} and tamarind pod shell powder after all experiments shows an adsorption band of 3396.79 cm^{-1} at 1.811 per unit area. This indicates that tamarind pod shell modified with KOH and powder obtained after experiments become more able to adsorption of light in a smaller surface area with the help of strongest O-H bond available and hence a lot of area is remaining for adsorption in the case of tamarind pod shell powder mixed with KOH and powder obtained after experiments which proves that adsorption can be increased with the increase in surface area.

D Analysis of rubber seed shell powder

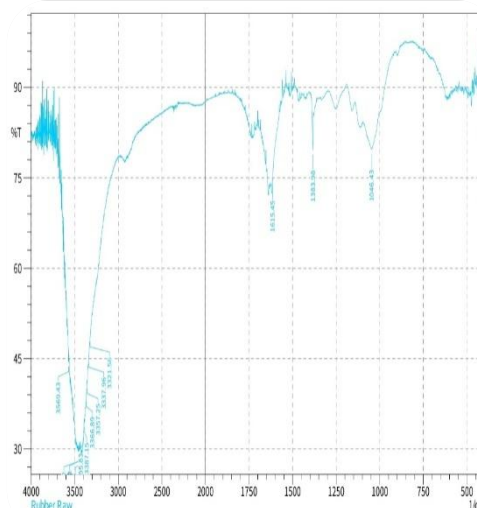


Fig 10 : rubber seed shell raw

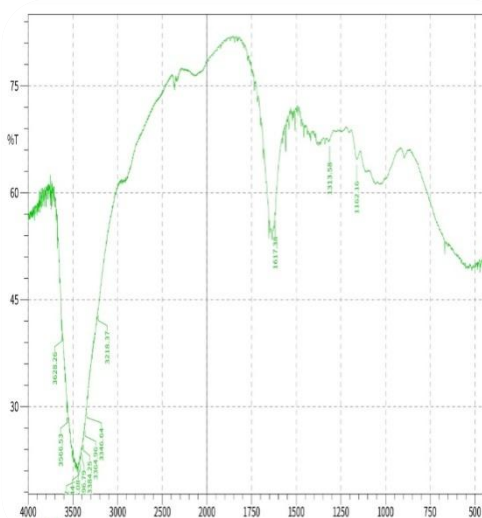


Fig 11: rubber seed shell KOH

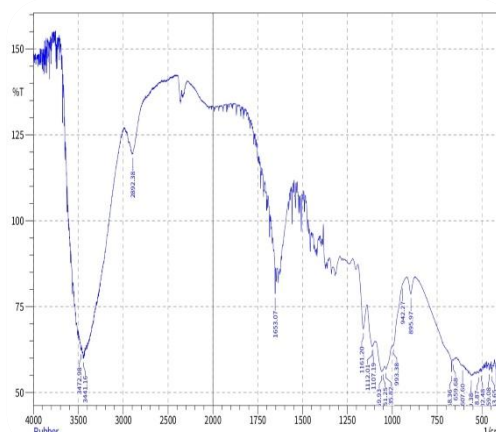


Fig 12 : rubber seed shell

FTIR of rubber seed shell raw powder dipped in KOH and powder after experiment are given below in fig 10 ,11 ,12 .In FTIR graph we have more than 5 peak so we can conclude as it is a complex compound. The sharp peak of rubber seed shell powder dipped in KOH shows sharp peak at 3628.26cm^{-1} and for powder after experiment sharp peak is at 3472.92cm^{-1} . We have more than 5 peak so we can see it as a complex compound.

There is no broad adsorption band in range between 3650 and 3250cm^{-1} that indicate the absence of hydrogen bond. There is peak in this area. It shows the sharp intensity of adsorption. It allows the compound to contain an oxygen related group, such as alcohol or phenol. We can see narrow band above 3000cm^{-1} shows aliphatic compounds. There is no peak between 2700 and 2000cm^{-1} so we can conclude there is no triple bond region inform the absence of carbon triple bond in material. To identify the double bond region we need to check between 1500 - 2000cm^{-1} . We don't have any sharp peak at 1750 - 1700 so it shows the absence of carbonyl groups.

In peak 1670 - 1620cm^{-1} shows the presence of unsaturation bonds specially we have peak at 1650cm^{-1} , it is for double bond carbon or olefin compound. The peaks between 1500 - 600cm^{-1} shows fingerprint region. In 1615.45cm^{-1} we have a sharp peak it inform about aromatic compound. In raw rubber seed shell powder at peak in 1046.43cm^{-1} shows an area of 4.275 per unit area. For rubber seed shell powder dipped in KOH at peak 1162.16cm^{-1} have area of 8.638, and for rubber seed shell powder after experiment have area of 10.5 per unit area at peak 1035.82cm^{-1} .

X. CONCLUSION

The journal can be concluded by analysing the impact of Iron removal by using modified low cost adsorbents. So the selected adsorbents by studying some journals such as Rubber seed shell, Pistachio shell, Groundnut shell and tamarind shell have been modified by KOH. Modifying by KOH increases the adsorption efficiency of adsorbents. This has been proved by FTIR analysis. The comparison of raw type, powder form modified by KOH and powder after the all experiments for each type adsorbents shows the adsorption efficiency is higher rate for powder with KOH and powder after all experiments. The efficiency comparison of each adsorbents have been done for the selected values of adsorption depending factors. Values taken from the journals which has been analysed using Minitab and optimization of values have been done. For this optimized conditions the efficiency of Pistachio shell is more than other shells.

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