Synthesis, Spectroscopic Characterization of Activated Carbon and Unactivated carbon derived from Limonia Acidissima Fruit Shell

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***Abstract*- The present work comprises the study of fine powdered activated and unactivated carbon of Limonia acidissima also known as Kavat shell (KSAC) impregnated by KOH used as an adsorbent. Physico-chemical characteristics of prepared activated carbon such as bulk density, porosity determination, moisture content determination, ash Content, and water-soluble matter, have been studied to assess the suitability of carbon as an adsorbent. Langmuir, Freundlich, and Temkin models were used to describe the isotherm and energy of adsorption. Spectroscopic Characterizations were studied to analyze the internal structure of carbon by Scanning Electron Microscopy, X-ray Diffraction which provides information about the arrangement of crystals and Fourier Transform Infrared Spectroscopy has been used to detect functional groups present in the sample.**

***Keywords*- Adsorption, impregnate, activated carbon, Physico-chemical Characterizations, Kavat shell activated carbon (KSAC)**

Introduction

Industrial waste, agricultural waste(biomaterial), and plant material are available in huge amounts biodegradable or non-biodegradable in nature inexpensive and environmentally friendly, and disposable. Pollution by heavy metals is becoming a serious global problem as they are degradable toxic and bioaccumulative [(1)]. Adsorption is a phenomenon widely used as an effective physical method for lowering the concentration of a wide range of dissolved pollutants organic and inorganic respectively. Activated carbon is the amorphous form of carbon specially treated to synthesize a highly internal pore structure and large surface area, an extremely versatile adsorbent of major industrial significance. It is commercially produced from bituminous or lignite coal, which is environmentally friendly and cost-effective. Activated carbon also plays an important role in decaffeination, gold purification, gas purification, water purification, metal extraction, sewage treatment, air filters in gas masks and respirators, filters in compressed air, and many other applications [(2)].

Heavy metal contamination is considered one of the important problems because of its harmful effects on humans and other substances. Adsorption is also known as the percolations process which is used in industry for purification and separation of solutes from fluid stream onto a surface [(3)].

Materials and Methods

Kavat Shells are collected from the local market and removed the pulp, washed with distilled water, and sun-dried for three days to remove impurities dried at 105^0 c for 24 hours in a hot box oven and then carbonized at 400^0 c for 1 hour in a muffle furnace (i-therm AI-7981). The carbonized sample cool and crushed with mortar and ground with an electric grinder. The sample was Sieved by 250 MICS (Standard test Sieve). Activated carbon was prepared by first impregnating Kavat Shells in $ZnCl_2$ solution followed by carbonization. Concentrations such as 20%, 40%, 60%, 80%, and 100% were prepared by dissolving $ZnCl_2$ in distilled water.

1.1. Adsorbent characterization

The physicochemical properties of the activated carbons such as the bulk density, porosity, moisture content, ash content, water-soluble matter, surface area, volatile matter, and fixed carbon content were determined by using standard procedures.

Parameters	20%	40%	60%	80%	100%	Unactivated carbon%
Bulk density g(cm^3)	2.62	2.68	2.73	2.75	2.77	2.69
Porosity cc/gm	0.05346	0.05469	0.05571	0.0561	0.05653	0.05489
Moisture content %	6	5	6	5.0	6.5	8.5
Ash content%	12.0	11.0	10.0	9.0	9.0	7.0
Water soluble matter(gm)	0.3	0.4	0.4	0.5	0.6	0.6
Surface area(m^2/g)	1,458.84	1479	1520.92	1556.44	1562.2	1267.48
Volatile matter %	46.80	44.73	46.80	46.52	46.80	45.35
Fixed carbon content	35.2	38.77	37.2	37.98	38.2	41.65

Table No-1

Metal analysis

To estimate the percentage removal of Co(II) from solution, the following equation was used.

$$\text{Percentage removal of Co(II)} = \frac{Cinitial - Cfinal}{Cinitial} \times 100$$

Where, C_{intial} and C_{final} are concentrations of Co(II) at the begining and at the end of adsorption process. The metal uptake (qe) at equilibrium time was calculated from the following equation

$$qe = \frac{(C0 - Ce)V}{100w}$$

Where qe (mg/g) is the amount of Co(II) adsorbed per unit weight of adsorbent, C_O and Ce are initial and equilibrium time lead ion concentration(mg/L), V is volume of solution (ml) and w is adsorbent weight (g).

Effect of Contact time

The effect on removal of cobalt studying when adsorbent 1g, 1.5g, 2g, 2.5g, and 3g kept constant and varied the metal ions solution concentrations 0.5N after respectively. The experiment was carried out at contact times 24hr. Figure 1- indicates that 20% activated carbon and unactivated carbon showed the same percentage removal of Co(II).

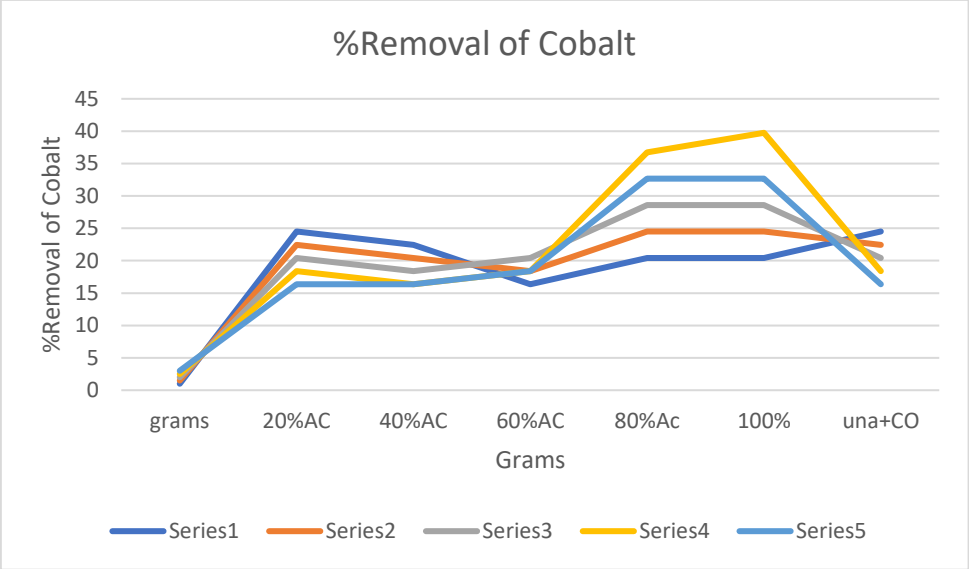


Figure 1 – % Removal for activated carbon of 20% ,40% ,60% ,80% ,100% and unactivated carbon

Effect of pH

The influence of operating time 24hrs. respectively. It is clear from figure-2. 20%,40%,60%,80%,100%AC and impregnated with 0.5N metal ions solutions shows higher pH (5.41-5.46) for 40% activated carbon and unactivated carbon.

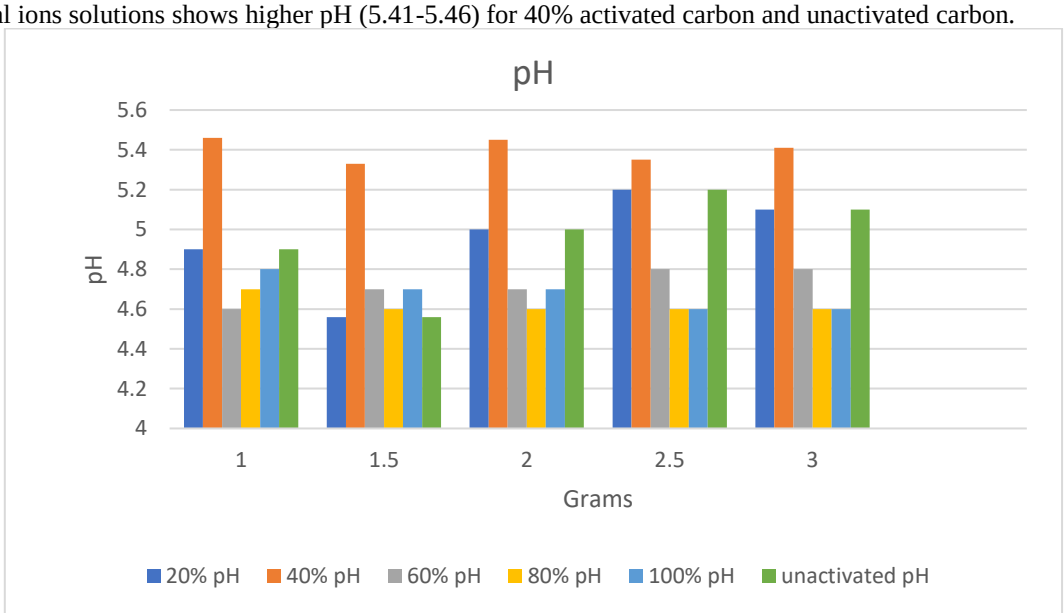


Figure 2.-Effect of pH on percentage removal

Result and Discussion-

Langmuir Isotherms adsorption

Carbon	Intercept	Slop	q_{max}(mg/g)	KL	RL	R^2
20% AC	2.0091	0.0066	0.497735	75.41444	7.938128	0.9271

40% AC	1.9894	0.01013	0.502664	49.62133	5.565163	0.9543
60% AC	2.1967	-0.02058	0.455228	-22.1199	-1.03503	0.2987
80% AC	2.07866	-0.00223	0.481079	-215.731	-18.8472	0.58505
20% AC	2.07866	-0.00223	0.481079	-215.731	-18.8472	0.58505
Unactivated	2.0091	0.0066	0.497735	75.41444	7.938128	0.9271

Freundlich Isotherms adsorption

Carbon	Intercept	Slop	1/n	K_f	R^2
20% AC	-0.2934	0.02319	0.02319	0.508862	0.9006
40% AC	-0.2541	0.09892	0.09892	0.557057	0.94062
60% AC	-0.3699	-0.06815	-0.06815	0.426678	0.3255
80% AC	-0.32606	-0.01315	-0.01315	0.471998	0.7243
100%AC	-0.32606	-0.01315	-0.01315	0.471998	0.7243
unactivated	-0.2934	0.02319	0.02319	0.508862	0.9006

Temkin Isotherms adsorption

Carbon	Intercept	Slop	$B_T(Jmol^{-1})$	$K_T(Lmg^{-1})$	R^2
20% AC	0.5083	0.01127	0.01127	3.87E+19	0.9012
40% AC	0.5172	0.0164	0.0164	4.97E+13	0.9443
60% AC	0.4229	-0.033	-0.033	2.72E-06	0.3243
80% AC	0.4718	-0.00639	-0.00639	8.6E-33	0.7257
100% AC	0.4718	-0.00639	-0.00639	8.6E-33	0.7257
Unactivated	0.5083	0.01127	0.01127	3.87E+19	0.9012

SEM Analysis- Prepared activated carbon was examined by scanning electron microscope (SEM) to analysed the surface of the adsorbents. The well-developed porous surface was observed in activated and unactivated carbon. ZnCl2-impregnated carbon consists of more canals-like structures than untreated carbon. SEM monograph shows that a wide variety of pores is present in activated carbon.

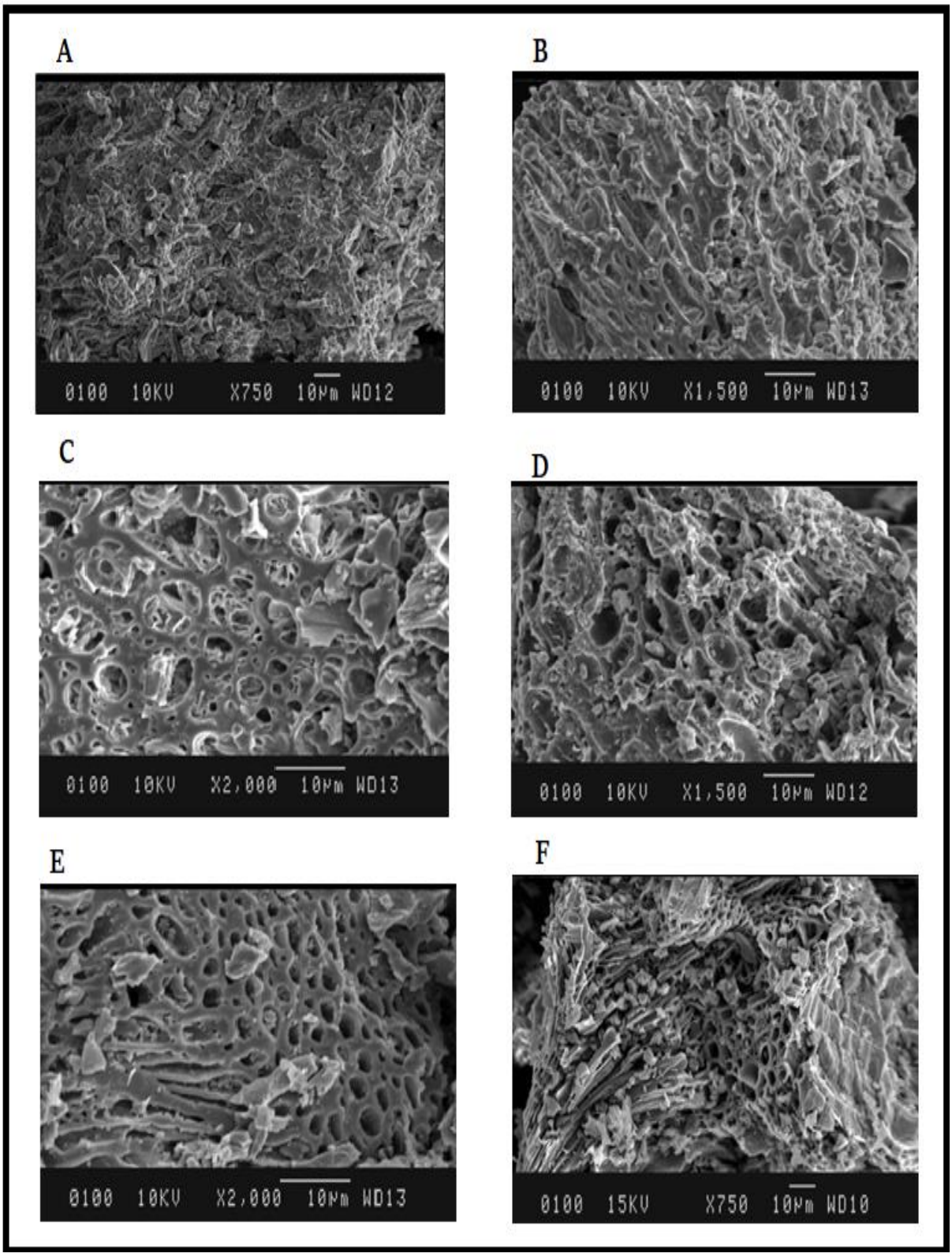
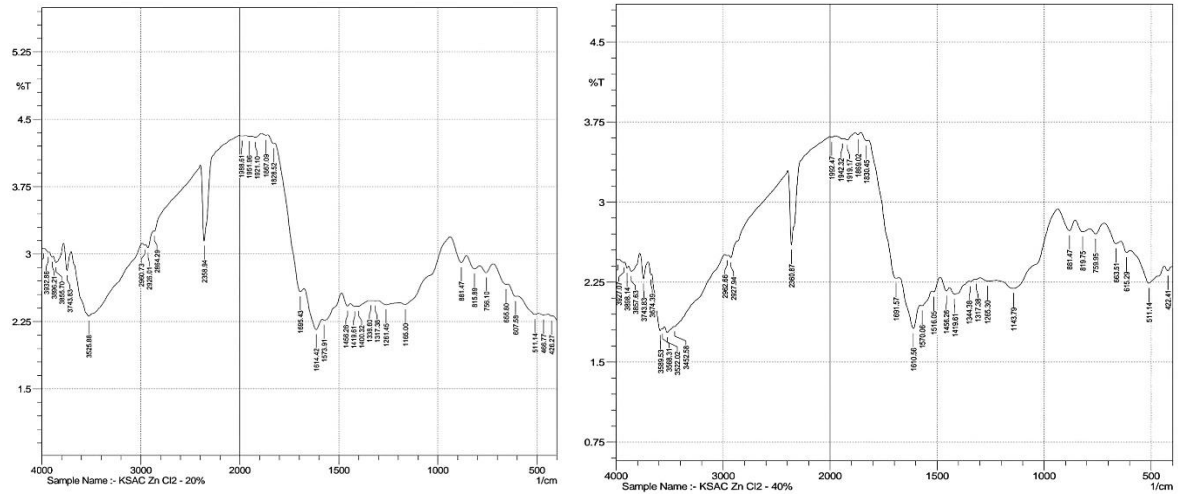


Figure 3. SEM monographs of $ZnCl_2$ AC A)20% B)40% C)60% D)80% E)100% and F) unactivated carbon

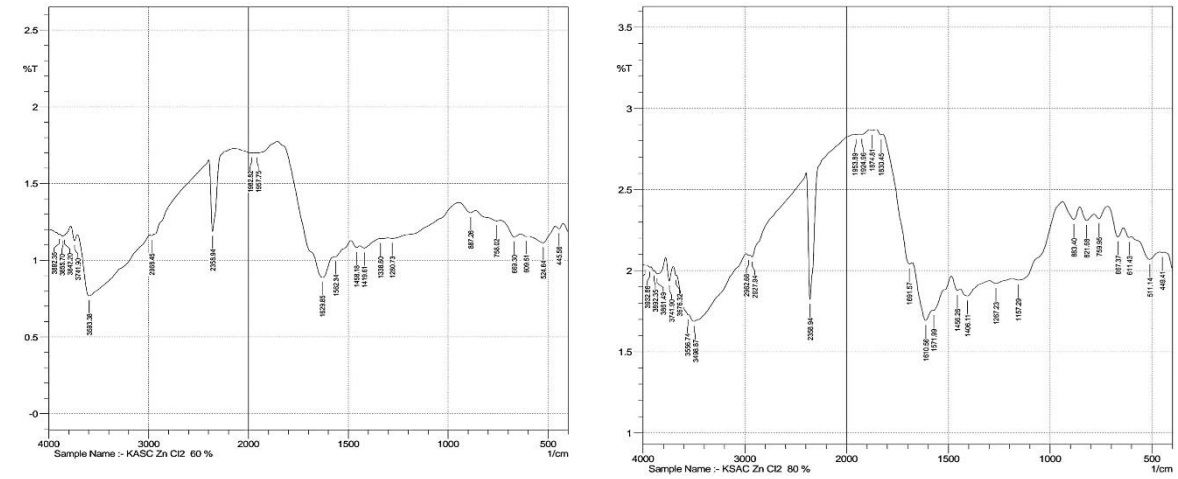
FTIR Analysis-

FTIR spectra showed the presence of oxygen-containing surface functional groups in the precursor. The FTIR band in the range of 3600-3500 cm^{-1} can be attributed to the O-H stretching vibration of hydroxyl functional groups in 20%, 40%, 60%, 80%, and 100% concentrations of activated carbon, and unactivated carbon also.

A) B)



C) D)



E) F)

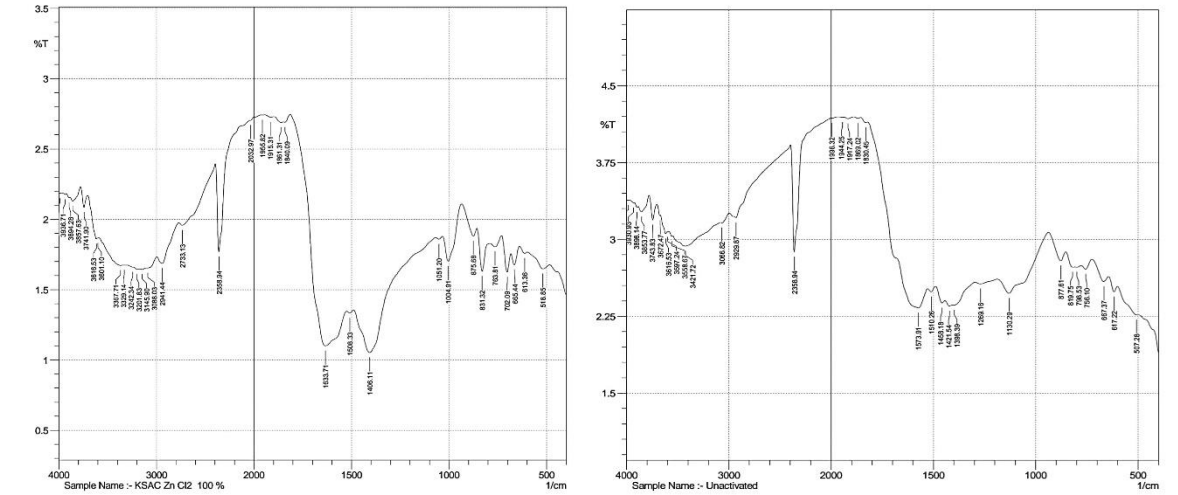


Figure 4.- FTIR monographs of $ZnCl_2$ AC G)20% H)40% I)60% J)80% K)100% and L) unactivated carbon

XRD Analysis –

X-ray diffraction spectroscopy (XRD) figures-5 analysis carried out with XRD-6100/7000 X-ray activated carbon prepared from Limonia acidissima shell by activating agent $ZnCl_2$ diffractometers Shimadzu. The activated carbon of Limonia acidissima 20% exhibited peak 2 theta= 20 ^{0}, 40% peak 2theta=28.9^0, 60% peak 2theta=24.07^0 and 48.8, 80% peak 2theta=20^0, 100% peak 2theta=27.06^0.29.45^0,30.09^0,34.08^0 . A comprehensive study was performed on its adsorption efficiency for the removal of transition elements. XRD analysis proved that the sample has a perfect amorphous structure.

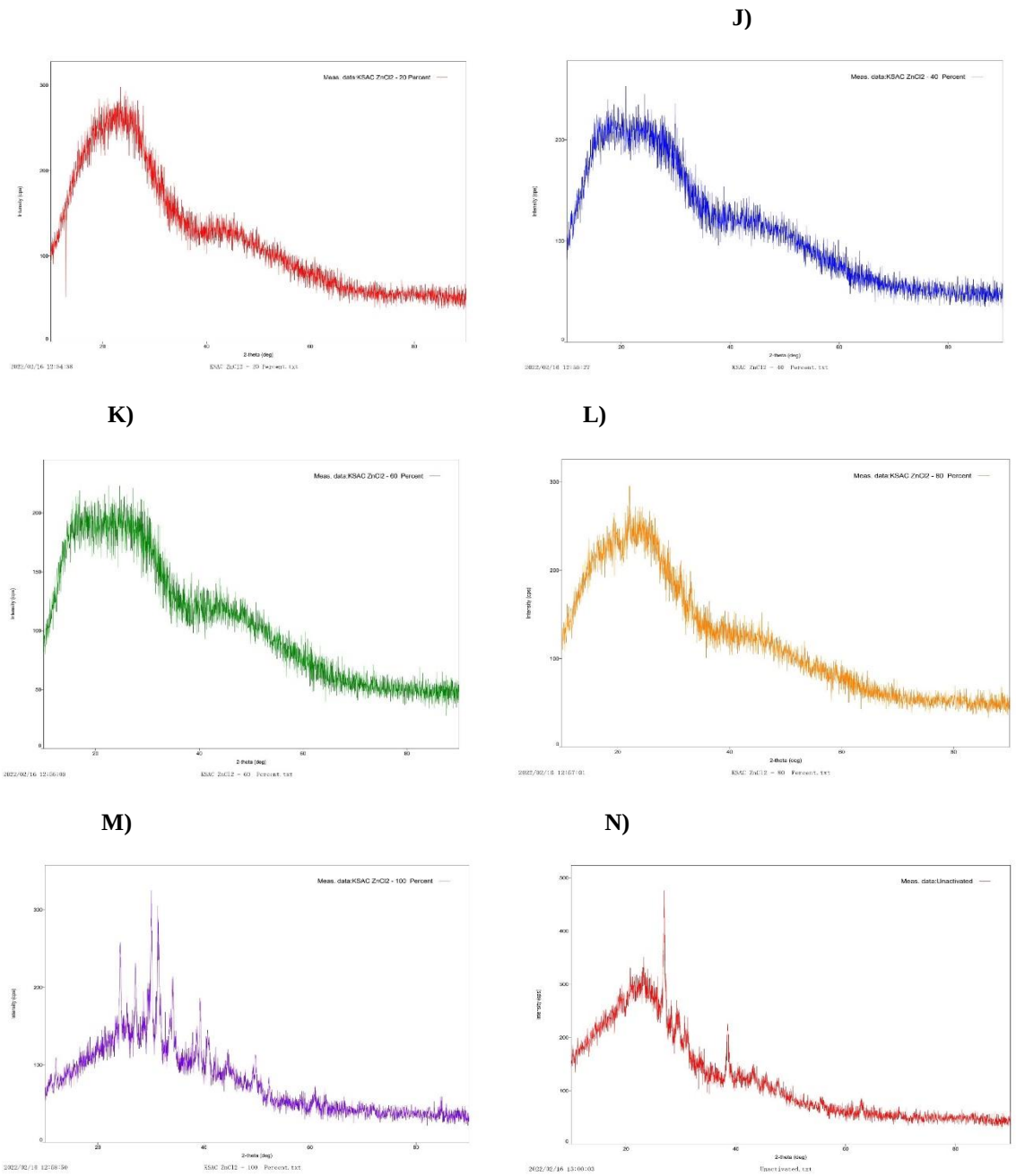


Figure 5.- XRD monographs of $ZnCl_2$ AC I)20% J)40% K)60% L)80% M)100% and N) unactivated carbon

Conclusions

Adsorption capacity is dependent on the activation process instead of a source of the raw material. The finding in this work suggests that the unactivated and 20% KSAC prepared by chemical activation using $ZnCl_2$ can be used as an effective adsorbent to remove Co(II) ions from aqueous effluent. The prepared activated carbon was characterized for bulk density, porosity, moisture content, Ash content, water-soluble matter, and percentage yield (Table No.-1) and used for removal of Co(II) by adsorption phenomenon. The activated carbon was also further characterized by XRD, FTIR, and SEM (figure: 3-5). The equilibrium data were well described by typical Langmuir, Freundlich, and Temkin, adsorption isotherms.

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