STUDY OF TOXIC HEAVY METAL REMOVAL BY DIFFERENT CHITOSAN/HYACINTHS PLANT COMPOSITE

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ABSTRACT

Water pollution with toxic heavy metals represents a most important problem, as a result of releasing these metals to ecological ambient without treatment, leading to their persistence and non-biodegradation. A variety of traditional methods are utilized as an attempt to remove heavy metals from waste water, but still without making any actual progress. In present work *Eichhorniacrassipes* (water hyacinth) root and shoot powder ,and ash case with chitosan are used as a removal system for heavy metals (HM), including lead, copper and cadmium from waste water via biosorption process .It was carried out by using two different experimental conditions: (i) heavy metal concentration (1000 ppm), with different pH values, ranged between 3-8 with a constant (HM) (ii) using variable (HM) concentrations with a range (250, 500, 750 and 1000 ppm). The results proved a good absorption for the hyacinths plant to the heavy metal ions especially chitosan /hyacinth shoot . Moreover, it was seen that highest removal (98%) of Pb (II), while was removed (98.2%) of Cu (II) and removed (96.8%) of Cd (II) where removal within 24 hr. On the other hand, FTIR spectra of composite materials showed only the characteristic of raw materials bands, which provided a strong indication of composite formation.

Keywords: Heavy metals, Eichhorniacrasspes, Biosorption, Ash ,Composite.

دراسة إزالة سمية المعادن الثقيلة بواسطة متراكبات مختلفة لنبات زهرة النيل مع الجيتسون

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المستخلص :

يمثل تلوث المياه بالمعادن الثقيلة السامة من أهم المشاكل، نتيجة لتحرر هذه المعادن إلى بيئة إيكولوجية دون معالجة، مما يؤدي إلى استمرارها وعدم التحلل البيولوجي. يتم استخدام مجموعة متنوعة من الطرق التقليدية كمحاولة لإزالة المعادن الثقيلة من مياه الصرف الصحي ولكن دون تحقيق أي تقدم حقيقي. في العمل الحالي يتم استخدام نبات زهرة النيل مسحوق (الجذر و الساق) وحالة الرماد للزهرة النيل مع الجيتسون كنظام إزالة للمعادن الثقيلة ،التي تتضمن الرصاص والنحاس والكادميوم من مياه الصرف الصحي عبر عملية الإزالة البيولوجية، وقد نفذت باستخدام نوعين من المعاملات المختلفة مع مختلفة تركيز ، مع قيم مختلفة الأولى تضمنت تركيز ثابت المعادن الثقيلة (1000) جزء في المليون مع قيم مختلفة من الاس الهيدروجيني تراوحت بين 3-8 بينما تمثلت الثانية باستخدام تراكيز متغايرة للعناصر الثقيلة ضمن المدى (250 ، 500 ، 750 و 1000 جزء في المليون). أثبتت النتائج امتصاص جيد لنبات زهرة النيل لأيونات المعادن الثقيلة وخاصة متراكبة الجيتسون مع ساق زهرة النيل . وعلاوة على ذلك ، لوحظ أن أعلى إزالة (98 ٪) من الرصاص (الثنائي) ، فيحين تمت إزالة (98.2 ٪) من النحاس (الثنائي) وإزالة 96% من الكادميوم (الثنائي).ومن ناحية اخرى اظهرت اطياف الاشعة التحت الحمراء خصائص أواصر المواد التي أعطت دليل قوي على تكوين المتراكبة ..

الكلمات المفتاحية: المتراكبة' رماد زهرة النيل' عملية الامتصاص ' زهرة النيل' العناصر الثقيلة.

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INTRODUCTION

The tremendous increase in the use of heavy metals over the past few decades has inevitably resulted in an increased flux of metallic substance in aquatic environment. Industrial wastewater contains higher amount of heavy metals that can pollute the water when it is discharged to the nature. Toxic heavy metals of particular concern in treatment of industrial wastewaters include zinc, copper, nickel, mercury, cadmium, lead and chromium. Heavy metals are the elements that have more than 5 times the specific gravity than that of water. Heavy metals are one of the most toxic types of water pollutants. At least 20 metals are considered to be toxic and approximately half of these metals are emitted to the environment in quantities that are risky to the surroundings, additionally to the human health. Wastewater containing heavy metals originated mainly from metal plating facilities, mining operations, fertilizer industries, tanneries, batteries, paper industries and pesticides galvanizing plants, stabilizers, thermoplastics, pigment manufacture, etc (1). These industries discharge heavy metals and wastewater directly or indirectly into the environment especially in developing countries. Due to their toxicity and non-biodegradability, they tend to accumulate in living organism. Therefore they cause numerous diseases and disorders. Zinc, copper, nickel, mercury, cadmium, lead and chromium are considered as toxic metals of particular concern in wastewater treatment. Thus, treatment of industrial wastewater containing soluble heavy metals has become essential in order to increase the quality of water Copper as an essential element plays an important role in all living organisms. It also widely used in industries such as high electrical and thermal conductivity, good corrosion resistance, ready availability, high recyclability and attractive appearance (2). Copper(II) is one of the heavy metals most toxic to the living organisms and it is one of more widespread heavy metal contaminants of the environment. Extensive intake of Cu can causeshemolysis, hepatotoxic and nephro toxic affects vomiting, cramps, convulsions, or even death (3). Recently, a number of studies were carried out on low cost adsorbents for removal of heavy metals from natural resources. One of such kind is the waste produced from fishery waste. Chitosan is a low cost adsorbent which is biodegradable and biocompatible polymer and it is produced by deacetylation of chitin (4).Chitosan is a unique basic polysaccharide and partially deacetylated polymer of glucosamineobtained after alkaline deacetylation of the chitin. It consists of mainly of β-(1-4-2- acetamido-2-deoxy-D-glucose) units and is the second most abundant biopolymer on earth after cellulose. It is widely distributed in crustacean shells and cell wallsof fungus (5). Chitosan is soluble in dilute acids. The solubilisation occurs by the protonation of the –NH2 function on the C-2 position of the Dglucosaminerepeat unit, where the polysaccharide is converted to a polyelectrolyte in acidic media (6) . Chitosan is the only natural cationic polymer and thus it is used in many applications for the wastewater treatment (7).The interest of study has focused on aquatic macrophytes such as water hyacinth (*Eichhorniacrassipes)*as promising candidates for pollutant uptake and good bioindicator of water pollution by heavy metals in aquatic systems **(**8**)**. Water hyacinth is one of the plant species that attracted considerable attention because of its ability to grow in heavily polluted water and also its capacity for metal ion accumulation (9)**.** Although there are other conventional methods such as primary treatment via physical sedimentation mechanism and chemical treatments that involve precipitation, adsorption and ion-exchange mechanism through adding of aquatic macrophytes treatment systems have more advantages as they require little energy for operation and they are capable of removing spectrum of contaminants (10). Aquatic macrophytes treatment systems are also free from usage of chemicals that are toxic both to humans and environment (11)**.** The overall objective of this study was to investigate that the heavy metals removal from wastewater by adsorption using hyacinth plant as a ash powder or with chitosan as composite under different pH degrees.

MATERIALS AND METHODS

Water hyacinth plant was collected from Tigres river in Baghdad . Chitosan of low molecular weight was supplied from ABCO

Laboratories(Eng. Ltd., Gillingham, England),acetic acid was provided by Fluka Chemicals Company (Switzerland). Sodium tripolyphosphate, (STPP) $Na_5O_{10}P_3$, molecular weight(367.86) and HCl was supplied from Mallinckrodt. Inc. (Paris, France) .$Pb(NO3)_2$, $CuSO_4$ $CdCl_2$,are the salts used in the preparation of stock standard solution (1 g/L) of analytical grade and HNO3, NaoH from Merck (Darmstadt, Germany).

Preparation of hyacinth plant powder

Plant parts was washed with distilled water for several times and divided into two parts (root and shoot).The root and shoot were dried at (105 ºC) for 24 hr and milled in a bladed mixer to less than 0.1mm and the powder was treated with 2M HCl for 24 hr . The powder washed several times with DW to reach pH 7. Thereafter , the powder was dried and milled again and stored in nylon package at room temperature until used.

Preparation of ash hyacinth plant

After washed Plant parts with distilled water for several times and divided into two parts (root and shoot); the root and shoot were dried at (210 ºC) for 3h,then following the same steps that used in method preparation of hyacinth plant powder, the only different in temperature and time to obtained ash hyacinth plant.

Preparation of chitason beads and plant ash

One gram of chitosan was dissolved in 40 ml of diluted acetic acid (2% v/v). Plant ash (root or shoot) was added as drops , into the chitosan solution during stirring process for 2 hrs and the mixture was preserved with ultrasonic bath for 30 min. The solution was plunged through a capillary tube into a beaker continuous 100 mL of STPP solution at pH 8.6. The solution of chitosan was dropped into the STPP solution and take shape spheres ,which are formed instantaneously. The hardened spheres were filtered; washed , dry and kept in a dry container for future used

Testing the abilty of samples in the removal of heavy metals in aquatic solutions preparation of stock solutions for sorption experiments: Standard solution of lead, copper and cadmium ions, were prepared with 1000 ppm concentration by dissolving 1.5 g,4.1g and2.25g, of the following heavy metal salts respectively which include lead nitrate, copper sulfate and cadmium nitrate, respectively in one liter of tris base buffer which is prepared in deionized water. These solutions were prepared using a standard flask, the range of concentrations used were prepared by serial dilution of stock solution with deionized water (12).

Batch sorption experiments

30 mg from each sample (powder and ash)from root and shoot hyacinth plant and 250 mg from beads composite sample in 50 ml of different heavy metal concentrations .The mixture agitated by shaker incubator for 30 inundate 35ºC.After each adsorption run, the mixture (adsorbent and adsorbate) was left for 24 hr and filtered by Whatman filter paper. Then, the samples were centrifuged at 3000 rpm for10min. The heavy metals concentration was determined by atomic adsorption spectrophotometer (13) .The removal efficiency (RE) of adsorbate (heavy metal) by adsorbent water hyacinth leaves was calculated by using the following equation (14):

$$\textbf{Removal \%} = \mathbf{Ci - \frac{Cf}{Ci} * 100\%}$$

Ci and Cf are the initial and final concentration respectively and measured by mg/L or ppm

Influnce of metal concentration

In order to analyze the adsorption potential of adsorbent in different concentration of the adsorbate, the following concentrations were prepared from the stock solution of each adsorbate (heavy metals) (HM) which include (250,500,750,1000 ppm).Throughout all experiments of these four concentrations, the pH value was adjusted at 7 which match the pH of water from which the plant was collected. The residual metal concentration was determined by atomic adsorption spectrophotometer. and calculated as in above.

Influnce of pH

The effect of pH on the percentage of Pb^{2+},Cu^{2+}, and Cd $^{2+}$,the pH of the solutions were varied from pH 2 to pH 8 using nitric acid and sodium hydroxide adjust pH condition of each one of these solution in aid of pH meter (Professional Bench top pH meter),then the same batch method performed to estimate up take capability of adsorbent in these divers pH conditions. The samples were stirred at a speed of 150 rpm for 2 h. The removal percentage of Pb^{2+} ,Cu^{2+} and Cd $^{2+}$

were determined in different acid and alkaline solutions , in the presence of nitric acid or sodium hydroxide. The pH degrees (2-7) were examined at 25ºC and 150 rpm for 2 hrs. The residual metal concentration was determined by atomic adsorption spectrophotometer, and calculated as in above.

Instrumentation

fourier transformation infrared spectrophotometer (FTIR): Fourier transformation of infrared (FTIR) spectra investigated were measured by Perkin-Elmer Fourier transformation of infrared spectrophotometer ((model 2000) over the wavenumber range of 400-4000 cm^{-1} in University of Baghdad /College of Education For Pure Science (Ibn-Al-Haitham).

Atomic absorption

The concentrations of Pb , Cu, Cd In altogether tasters were determined according to standard method .using Atomic Absorption Spectrometer Varian Spectra (220) with graphite furnace accessory and equipped with deuterium arc background corrector in University of Baghdad /College of Education For Pure Science (Ibn-Al-Haitham).

RESULT AND DISCUSSION

Root or shoot powders of hyacinth plant revealed different in their ability to remove heavy metals .The result proved that the root removing efficiency was higher than the removal ofCu and Pb metal ions when shoot applied, while there was no removal observe concerning Cd (Fig.1).The removal percentages of heavy metals had been effected according to the plant part powders. The results showed that the copper removal percentage is 80 and 70 %, while the removal of lead was 52 and 25 % when plant root and shoot powders were used, respectively. These were attributed may be to the amounts of heavy metals that absorbed during the plant life. Furthermore, the plant cell wall made of functional groups consisting of proteins, carbohydrates and lipids which act as binding sites for these interactions and accumulation of metals in plant parts [15,16].More specifically, the binding sites of (Pb) may not be the same as in other ions ,while its larger molecular weight leads to more paramagnetic and electronegative compared to other metal ions. The hydrolysis constant of copper and lead (Pb) are much greater, implying that the ionic properties could make it more favorable to sorption sites (17).

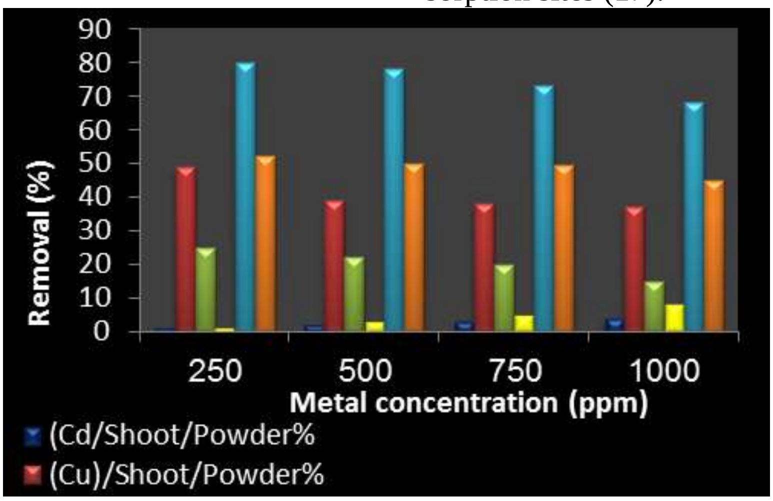

Figure 1. Heavy metals removed by 0.6 mg /mlroot and0.6mg/ml shoot powders of hyacinth plant

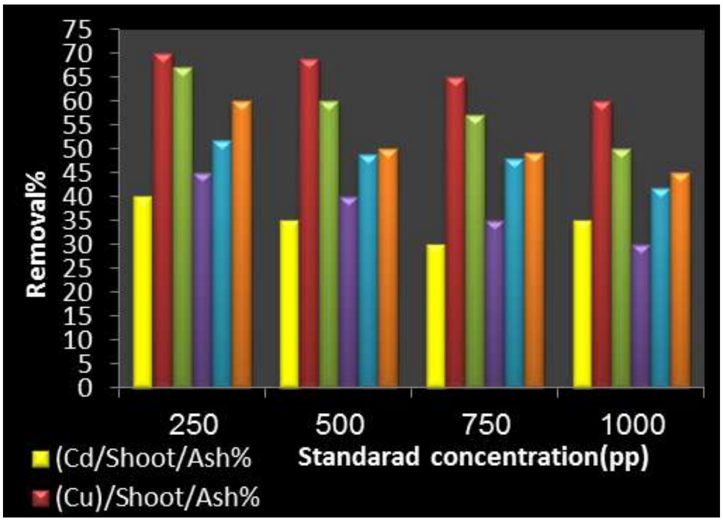

Figure 2. Heavy metals removed by 0.6 mg /ml root and 0.6 mg/ml shoot ash of hyacinth plant

Figure (2) shows the hyacinth root and shoot ash removal as a function to the standard concentration .The results found that the shoot ash was more effective than root ash in Cu ,Pb and Cd removal .The results showed that Cu^{2+}, Pb^{2+} and Cd^{2+} adsorption for (shoot and root) ash were better than that for (shoot and root) powder . It was seen that the shoot ash removal was more than that for hyacinth root ash .It was found that lead (pb) had the highest removal percentage (67.2%)Pb, (70%)Cu and (60%)Cd at low concentration (250 ppm). The result were attributed to physical adsorption ,which induced by Van der Waals forces between metal ions and ash particles. Ash contains negatively charged carbon particles, and metal ions are positively charged heavy metal's particles generally show higher adsorption capacity compared to lighter metals ; that is in a good agreement with [20]. Ash of water hyacinths plant contains oxides of Na, K, Ca, and Mg. These oxides are basic or amphoteric. Upon their reaction with water, they produce hydroxides. The pH of ash is increased due to the formation of these hydroxides. The following insoluble hydroxides are formed: Cd^{2+}, Pb^{2+} and Cu^{2+}, and due to low constant for solubility product. these are precipitated. The Precipitation or co-precipitation is also cause of adsorption onto ash of these metals. Based on the spontaneous nature of the sorption process, it is possible that mechanism of sorption may be ion exchange in nature. The most important components in the plant's ash which is oxides/hydroxides of Ca, Mg, K, Na, Al, Fe, and Si. These underlie phenomena such as adhesion, precipitation, co-precipitation, sequential precipitation, surface precipitation, and adsorption. Van der Waals forces may be responsible for physical adsorption, while precipitation and ion-exchange may be responsible for chemi-sorption. The pretreatment of ash with deionized water and adsorption results have also been verified by recent studies. The deionized water may remove soluble ions and create sites for attachment of metal ions. Figure (3) shows the chitosan \hyacinths plant (root and shoot)ash as function to standard concentration ;it was seen that chitosan\hyacinths plant shoot ash adsorption is superior than that of the root ash .the highest removal percentage Pb (98.2%) , (98%) Cu and (60%)Cd at low concentration (250 ppm).Cu removal was higher than that (Pb and Cd).

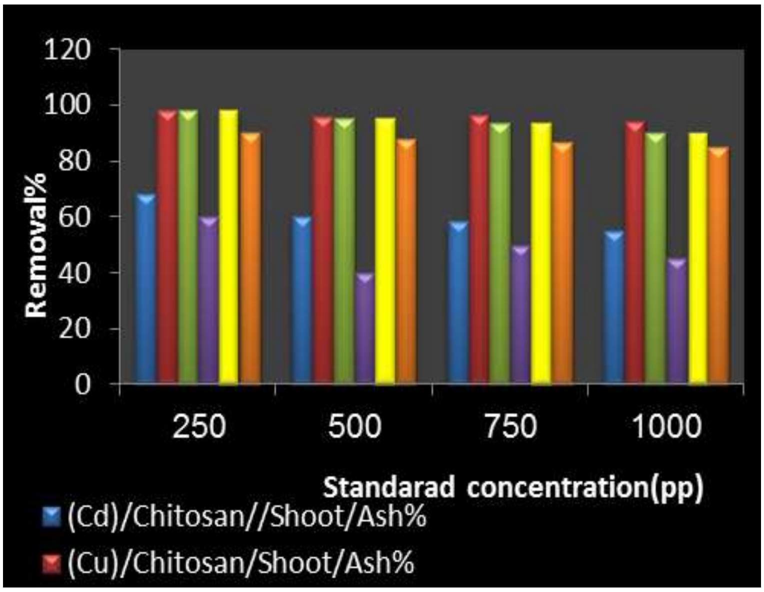

Figure 3. the removal of chitosan\ hyacinth root and shoot ash removal as a function of standard concentration(ppm).

Aquatic plants play a major role in monitoring or minimizing of contaminants including heavy metals in aquatic ecosystem. Many studies reported the importance of different water plants in their acting as bisorbent. (18). However, there are certain disadvantages of using living plants for bio sorbent purposes; such as its necessary to constructed artificial ponds near the intended point as well as the phototoxic effect of these ions which might result in inhibiting chlorophyll synthesis and necrosis (16). In contrast, moving and handling dead biomass is more advantageous compared to living form (19). Moreover, research have been done extensively on roots of these plants ,while little is known about its leaves (20). Tables (1-3) show the Pb, Cu, Cd removal as a function to the (PH). These results demonstrated that the hydrogen ion concentration (pH) influences the adsorption process (21). It was found that pH is one of the important factors that affect the solubility of metal ions through altering the positive sites occupied by protons on the adsorbent surface with negative charge when alerted from 3 to 8, therefore more binding sites are available for position ions. The results show the removal Pb by used (root and shoot) powder or ash hyacinth plant decrease with increase (pH) , but the removal by used composite chitosan and hyacinth plant (root and shoot) in form powder or ash increase with increase(pH), while the removal of (Cu and Cd) with (pH) inverse the removal Pb , its removal by used (root and shoot) powder or ash hyacinth plant increase with increase (pH) , but the removal decrease by used composite when increase (root and shoot) powder or ash hyacinth plant decrease with increase (pH), this fact proved by this fact proved by (22). The results show in table (1-3)

Tables 1. Removal of heavy metal percentage(Pb, Cu and Cd) (ppm) by using different part of hyacinth plant in case powder ,ash and with chitosan with variable pH value

Chitosan with root ash removal %	*Chitosan with shoot ash removal %*	*Chitosan with root powder removal %*	*Chitosan with shoot powder removal %*	*Root ash removal %*	*Shoot ash removal %*	*Root powder removal %*	*Shoot powder removal %*	*Type of Heavy Metal Ion*	*PH*
80	98	85	75	60	67.2	20	15	Pb	3
98.1	98.2	87	78	59	67	19	14	Pb	4
98.4	98.4	89	80	58.4	66	18	13	Pb	5
98.6	98.6	91	82	58	65.4	17	12	Pb	6
98.8	98.8	93	84	57.5	64.7	16	11	Pb	7
92	99	95	86	57	64	15	10	Pb	8
90	98.2	99	97	56	70	16	14	Cu	3
92	98.4	99.1	97.5	55	69	15	13	Cu	4
93	98.6	99.3	98	54	68	14	12	Cu	5
94	98.8	99.5	98.5	53	67	13	11	Cu	6
95	100	99.7	99	52	66	12	10	Cu	7
96	100	100	99.5	51	65	11	9	Cu	8
54	66	60	52	52	60	11	12	Cd	3
56	67	61	51.5	51	59	10	11	Cd	4
58	68	62	52	50	58	9	10	Cd	5
60	69	63	53	49	57	8	9	Cd	6
62	70	64	54	48	56	7	8	Cd	7
64	71	65	55	47	55	6	7	Cd	8

The characteristics of the hyacinth plant are shows,the band at (3365 cm^{-1}) was attributed to (OH) stretching vibration . The stretching vibration of (C-H) is at (2920 and 2925 cm^{-1}). The band at (1639cm^{-1}) was due to amide band, and the band at (1427cm^{-1}) was matches to (CH_2) bending at (CH_3) deformation ; The appearance of the band at (1030cm^{-1}) is due to (C-O) stretching band ; and band at (580 cm^{-1}) is attributed to (chloride , bromide , iodide. The results were in a good agreement with those in [1]. The band assignment of water hyacinth plant powder root and ash root is illustrated in Fig (4-7), the ash root hyacinth plant shows the bandFTIR spectra are assigned as follows: 3433cm^{-1},2941 cm^{-1} and 2885 $cm^{-1}$the bands increase than powder of root hyacinth plant and the 1709,1599 and1458$cm^{-1}$of C=C-C aromatic ring stretching; 1074 cm^{-1}aromatic C-Hstretch; 1028cm^{-1}C-O-C stretch associated with -OH bend cellulose, hemicelluloses and lignin; 895 and 820cm^{-1}aromatic C-H out-of-plane bends. The spectra of ash shoot hyacinth plant show in fig (4-8) higher intensities of aromatic absorption bands than ash root hyacinth plant , indicating an increase of aromatic C and better in removal heavy metal ions from waste water.

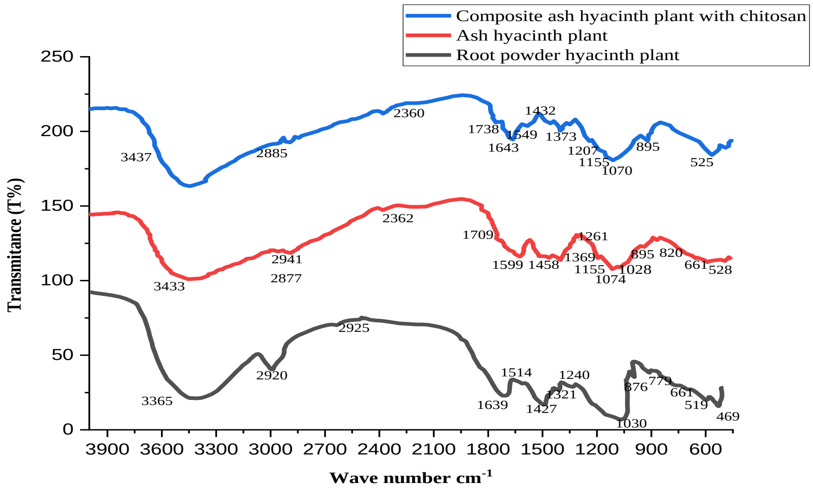

Figure 4.FTIR spectra of :(A) Root powder hyacinth plant ; (B)Root ash hyacinth plant and(C) Composite ash root hyacinth plant with chitosan

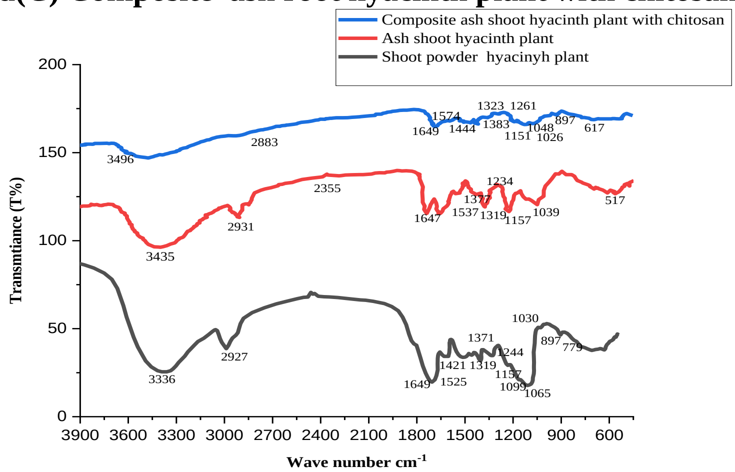

Figure 5. FTIR spectra of :(A) Shoot powder hyacinth plant ; (B) Shootash hyacinth plant and(C) Shoot ash hyacinth plant with chitosan

Fig (4,5) depicts the result that displays the FTIR spectrum of ash root or ash shoot water hyacinth/chitosan composite that include the typical bands of cross-linked chitosan respectively, root ash composite and shoot ash composite appearing at at(3437,3498) cm^{-1}; It was attributed to hydroxyl band stretching (-OH)and(-NH)groups . Appearance of band within (2883– 2885 cm^{-1}) that was related with C-H stretching vibration aldehyde, the (1643, 1649) cm^{-1} and(1549 ,1574) cm^{-1} matching to extending vibration of (P=O) of the (-POH)groups and extending vibration of (NH^{3+}) group correspondingly. In addition, the band displays the central peaks of water hyacinth dry substance which seems at (1373,1378) cm^{-1} and (1207, 1261) cm^{-1} which matches to CH_3 umbrella style. The nearby band nearby which agrees to CH3 umbrella mode. The band near(1155 and 1151) cm^{-1} is illustrative of the ant symmetric bridge extending of C-O-C groups in cellulose. After the overhead point out results it could be determined that there is no new bands give the impression in the spectrum of water hyacinth/ chitosan composite which provide strong indication that there are ready material is a composite in which offers the particles of (water hyacinths plant) are implanted in the matrix of cross-linked chitosan polymer.The spectra of all native bio sorbents revealed many absorption peaks in the 800 – 400 cm-1 region, which may be related to the high mineral (ash) content of the bio sorbents.This study showed that the ash of water hyacinths plant offers several advantages including cost effectiveness , high efficiency, minimization of chemical/biological sludge,and regeneration of biosorbent with possibility of metal recovery. The adsorption capacity of *E. crassipes*ash to metals is more than that of other plant materials. It was found that chitosan /hyacinths (root and shoot) ash of the best removal whereas the removal efficiency for the shoot was better than that for the root .It was conclude that these material satisfies demands for waste water remediation. Biosorption and desorption by the ash of water hyacinth is technique can be used for removal and recovery of metal pollutants from water.

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