Preparation of a Magnetic Composite Based on Cellulose Nanocrystals and Polyvinyl Alcohol and its Efficient Use for Removal of Nickel and Zinc Ions from Aqueous Solutions

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ABSTRACT

Heavy metals are harmful ions and their removal from wastewater has attracted a growing attention amongst the scientific researchers in current decade. In this study, *pea peels* were used as precursor to purify cellulose nanocrystals (CNCs). Polyvinyl alcohol/ CNCs magnetic composite was developed as a novel bioadsorbent for adsorption of Ni (II) and Zn (II) ions from aqueous solutions. Results revealed that the adsorption efficiency of the examined ions by PVA/CNCs magnetic composite largely increased with increasing contact time. The equilibrium adsorption capacity was 4.19 and 4.00 mg g^{-1} for Ni (II) and Zn (II), respectively. The highest removal percentages for Ni (II) and Zn (II) were 83.86 and 80.13, respectively at initial concentration of 10 µg/ml. The FTIR and SEM EDX analysis were performed before and after Ni (II) and Zn (II) sorption on the PVA/cellulose beads, validating that the -OH groups played a chief role in sorption process. Finally, it could be concluded that PVA/cellulose magnetic composite is a promising adsorbent for removing Ni (II) and Zn (II) ions from wastewater.
Keywords: *Pea peels*, cellulose nanocrystals, PVA, magnetic composite, heavy metals.

INTRODUCTION

Heavy metal contamination is considered as one of the most harmful effects on environment and human health as a result of their extreme toxicity, non-biodegradability and accumulation on food chain sequence organisms *(Nordberg et al., 2007)*. Metal-contaminated wastewater is generated yearly through various agricultural and industrial disposal processes *(Mark et al., 2006)*. Remarkably, heavy metals adsorbents based on agricultural wastes are gaining much attention in last two decades on account that they provide advantages such as low-cost, abundance, highly effective *(Mark et al., 2006)*. The main constituents of the agricultural waste resources include lignin, hemicelluloses, starch, sugars, proteins and lipids, containing different functional groups with a likely sorption capability for harmful metals (Bhatnagar *et al., 2015*). Cellulosic resources are being largely used as they deliver particular properties including non-toxicity, barrier function, edibility, biocompatibility, attracting appearance and low cost (*Imran et al. 2010*). Cellulose-based agricultural wastes are smoothly used in the removal of dangerous ions from wastewater. In this respect *UlHaq et al.* (2017) stated the efficacy of pea powdered peels to remove Pb and Ni from aqueous solutions. Different modified structures have been reported to increase sorption capacity of cellulosic wastes i.e. CNCs, biochars and magnetic composite (*Bhatnagar and Sillanpää 2010;* Khattak *et al., 2017; Ahmad et al. 2018)*. Biodegradable polymers such as polyvinyl alcohol (PVA) could replace polyethylene or polypropylene to supply environmentally satisfying product and biodegradable composite materials. PVA is a water-soluble material involving many active hydroxyl groups which contribute to its hydrophilic character. It exhibited a good film-forming property with brilliant physicochemical stability *(Koops et al., 1994)*. Interestingly, PVA/chitosan macro-porous has been reported to eliminate heavy metal from aqueous solutions *(Li et al., 2011)*.

Nickel (Ni) salts are the prevalent reason of metal allergy among the publics seemed as contact dermatitis *(Kasprzak et al., 2003)*. Zinc (Zn) is a critical element for a variety of life, but it is also highly toxic when used in high concentration. No available data in the literature about the usage of cellulose from *pea waste* in developing of a magnetic composite for the elimination of the previously mentioned harmful ions from wastewater. Therefore, the objective of this study is to prepare and characterize the magnetic composite resulting from the combination of PVA and pea by-product purified CNC, and to estimate the adsorption capacity of the resultant composite for the removal of Ni and Zn from aqueous solutions.

MATERIALS AND METHODS

Pea peels samples

Green *pea by-product* (GPP) was obtained from local market. *Peels* were washed with distilled water (D.W), air dried for 72 h and powdered to a uniform size.

Chemical purification of cellulose

Chemical purification of cellulose from *pea peels* was done using the scheme of *(Kumar et al. 2013)* with slight modifications as follows:-

Pea peels (50.0 g) were first de-fatted in a soxhlet unit using hexane for 6 h. The solid phase was then acidified by sodium chlorite solution for the removal of lignin at 75°C for 90 minutes; this step was repeated for 4 times until the residue became white. The white residue was treated with 3 % (w/v) NaOH at 85-90°C for 120 minutes followed by filtration to eliminate hemicelluloses and other polysaccharides. The product was finally washed with D.W until the filtrate became neutral. Cellulose was dried at 105°C for 6 h and stored until used.

Extraction of cellulose nanocrystals

Extraction of cellulose nanocrystals (CNC) was performed by acid hydrolysis according the method of *(Hudson et al., 2013)* with slight modifications. The hydrolytic procedure was completed at 45°C for 60 min under the aid of stirring. One gram of purified cellulose was added to 12 mL of H$_2$SO$_4$ 9.17 M. At the end of the reaction, cold water was added to stop the hydrolytic reaction; the suspension was centrifuged for 15 min at 6000 ppm to exclude the acidity. Then, the nascent precipitate was dialyzed against tap water until the neutral pH to remove soluble sugars, salts and non-reactive sulfate groups. Finally, the dialyzed suspension was considered as CNC of pea *peel*.

Preparation of PVA/CNC magnetic composite.

PVA (1g) was dissolved in distilled water (100 mL) at 70 ^{o}C with continuous stirring for 2 hour *(Zhu et al. 2014)*. Then CNC suspension was mixed with PVA solution and the blend was agitated again for 30 min. Cations of Fe^{++} (as FeSO$_4$) and Fe^{+++} (as FeCl$_3$) (0.01 mol: 0.02 mol) were supplemented into PVA/CNC mixture with continuous stirring for 40 min. Then the resultant solution was introduced dropwise into a conical flask holding 150mL sodium hydroxide solution (8%, w/v) to make the magnetic beads. Finally, solidification was performed by continuous stirring of the resultant alkali solution for 60 minutes, and then the beads were separated from the alkali solution and washed with D.W until pH ~ 7. The resultant composite was dried at 70 ^{o}C for 18 h.

Characterization of PVA/CNC magnetic composite

PVA/CNC magnetic composite was characterized by Vibrating Sample Magnetometer (VSM) (730T, Lakeshoper, America) (Hong *et al* .2008), Fourier Transform Infrared Spectroscopy (FTIR, Spectral Analysis Unit Chemistry Department Faculty of Science Mansoura University), scanning electron microscopy (JEOL JSM-6510 LV, Japan, nanotechnology center of Mansoura University) and thermogravimetric analysis (TGA) *(Ahuja et al., 2017)* and differential scanning calorimeter (DSC) derivative studies.

Adsorption experiment and Analytical methods

The trials were carried out at room temperature by shaking 40 mg of PVA/CNC magnetic composite with twenty mL solutions (10, 25 and 50 mg L^{-1}) for 30 h at 140 rpm and pH 6.0. After adsorption, the magnetic composite was removed by filtration. For the kinetic trials, the initial concentration of Zn (II) and Ni (II) was ten mg/L, the contact time was 5, 10, 15, 20 and 25h and the adsorbent mass was 40 mg. The concentrations of Zn (II) and Ni (II) were analyzed by atomic absorption spectrometer (Model SENS AA Dual, GBC Scientific equipments). The equilibrium adsorption capacity and the removal percentage can be calculated by the next equations:

$Q_e = (C_0 - C_e) V / W$ (1) Removal % = $[(C_0 - C_e)/ C_0] \times 100$ (2)

Where Q_e (µg/ml) is the equilibrium adsorption capacity; C_0 and C_e (µg/ml) are the initial and equilibrium concentrations of Ni (II) and Zn (II), respectively; V (L) is the volume of the Ni (II) and Zn (II) solutions, and w (mg) is the mass of magnetic adsorbent.

RESULTS AND DISCUSSION

Magnetic composite was initially prepared using PVA and the purified CNC and then described. Lastly, adsorption behavior of the resultant magnetic composite for removal of Ni (II) and Zn (II) from aqueous media was investigated. Fig 1 shows physical appearances of *pea peels* (A), chemically purified cellulose (B), cellulose nanocrystals (CNC, C), ferrous and ferric salts solution (D) and prepared PVA/CNC magnetic composite (E). The schematic proposed structure of PVA/CNC bead is presented in Fig.2. The interacting hydroxyl groups on the surfaces of both CNC and PVA yield hydrogen bonds *(Jayaramudu et al. 2018)*. Additionally, the higher quantity of CNC dispersed in the formed hydrogel, the higher is CNC aggregation as a result of the hydrophilic property of CNC, causing the uniformly rough surface of the resultant composite *(Jayaramudu et al. 2018)*.
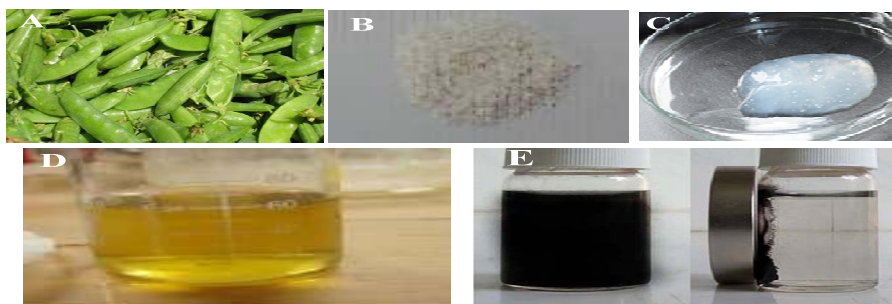

Fig. 1. Photo of (A) *Pea peels* (B) Chemically purified cellulose (C) Cellulose nanocrystals (D) FeCl$_3$ and FeSO$_4$ solution (E) Preparation the cross-linked of PVA/Cellulose nanocrystals magnetic composite.
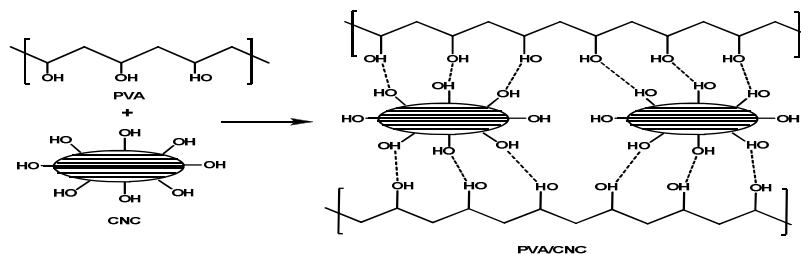

Fig. 2. Schematic proposed structure of PVA/CNC magnetic beads

Characterization of PVA/CNC composite Magnetic properties

To declare the magnetic property of the resultant nanocomposite as a utility of the applied magnetic field at 25 ^{o}C, the beads were examined using VSM .The saturation magnetization of PVA/CNC magnetic composite was calculated to be 19.748 emu g^{-1} (Fig 3), indicating that the resultant composite was super-paramagnetic due to the occurrence of considerable content of iron oxide. So, PVA/cellulose magnetic composite can be simply removed with the aid of a magnetic force. *Zhu et al (2014)* found that the saturation magnetization for PVA/chitosan magnetic composite was 21.4 emu g^{-1}.
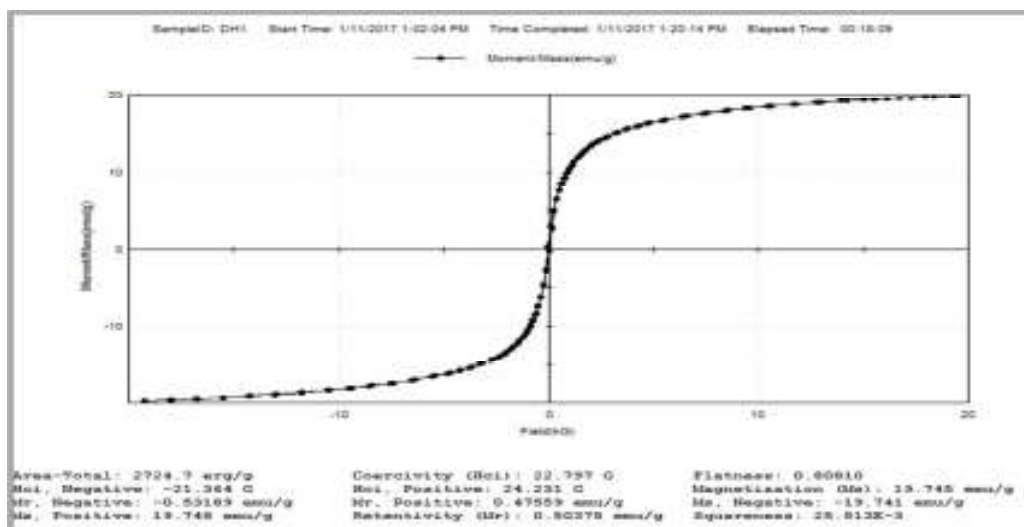
Fig. 3. Magnetic properties of prepared PVA/Cellulose magnetic composite

Thermogravimetric analysis (TGA)

TGA is a powerful procedure in estimating of the thermal stability of the materials *(El-Sayed et al., 2011)*. In this trail, PVA/cellulose nanocrystal magnetic composite was decayed by heat, which breaks the bonds in the molecules. The derivative DTG indicates the equivalent rate of weight loss. DTG max is the temperature at which the thermolysis takes place at maximum rate. Thermal stability of various materials such as PVA/Cellulose magnetic composite could be assessed by determining their values of DTG max. Fig.4 presented the weight loss pattern on thermal decomposition of PVA/CNC magnetic composite that following multi-step thermal decomposition system as shown by *(Rhim et al., 2013)*. TGA and DTG curves of PVA/Cellulose nanocrystals magnetic composite covered a temperature range from 120 to 650°C.The initial weight loss was around 240°C as a result of the vaporization of absorbed moisture. The degradation phase at 240 and 340°C relates to degradation of PVA. The last step of degradation (> 615°C) corresponds to cellulose thermolysis *(Elhefian et al., 2012)*.

Thermal stability studies on PVA/CNC magnetic composite were evaluated through DSC where heat of reaction was recorded. Enthalpy obtained from DSC (Fig 4) indicated good thermal stability for PVA/CNC magnetic composite. This shows that further energy is needed to break down the bonds *(Watkins et al., 2015)*.
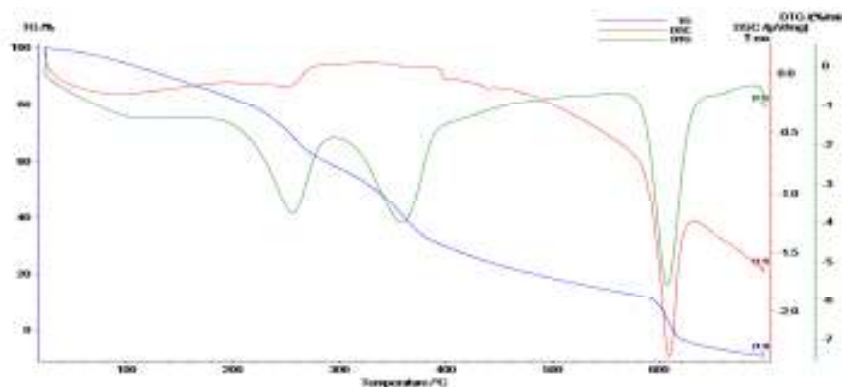

Fig. 4. TGA, DSC & DTG curves of prepared PVA/Cellulose nanocrystal magnetic composite

FTIR Spectral Analysis

For confirmation of the structure of the prepared PVA/CNC magnetic composite, FTIR spectra of PVA/cellulose magnetic was investigated before and after the adsorption of the examined ions (Fig, 5). The characteristic peaks of cellulose derivative are established as follows: the broad peak around 3418 cm^{-1}relates to -OH stretching vibration, the peak at 2927 cm^{-1} is qualified to -CH stretching vibration, the peak around 1628 cm^{-1} relates to of C=O. The bands in the range 1020–1100 cm^{-1} are assigned to pyranose skeleton *(Yang et al., 2009)*.The peaks at 530-660 cm^{-1} are assigned to the occurrence of Fe-O, showing that $Fe_3O_4$was successfully covered by PVA/cellulose nanocrystals. By matching the pictures before (Fig. 5A) and after adsorption (Fig. 5B), an extensive decline of adsorption intensity of the peak at 3418 cm^{-1} was clearly noticed. Peak shift was also observed from 3418 to 3424 cm^{-1}. This means that –OH group participates in the adsorption route. The peak at 1020 cm^{-1} matching C=O stretching was somewhat shifted to 1057 cm^{-1}. From the FTIR description of the PVA/cellulose nanocrystals beads, it can be established that -OH is the main contributor for the adsorption of Ni (II) and Zn (II).
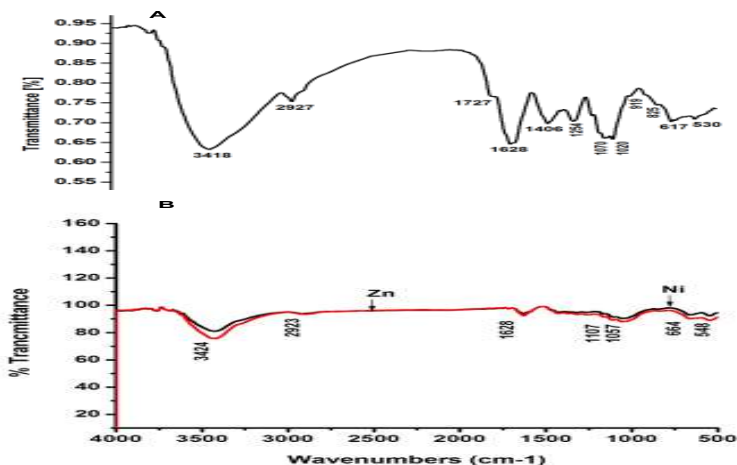
Fig. 5. The FTIR spectra of PVA/ CNC composite before (A) and after (B) Ni (II) and Zn (II) sorption.

The FTIR study of PVA/CNC magnetic composite before and after metal adsorption showed that the major complexing site is -OH, since the atom of oxygen in -OH has a pair of electrons that can be added to proton or cation by a coordinated covalent bond. Ni (II) and Zn (II) ions have empty orbitals (Lewis acid), that receive electron pairs. As a result, the coordination conceivably designed by electron transfer *(Zhu et al., 2014)*.

Table 1. The equilibrium adsorption capacity (Q_e) and percentage of removal (Removal %) of Ni (II) and Zn (II).using PVA/cellulose magnetic composite.

Metals	Initial concentration (µg/ml)	The concentration (µg/ml) after Adsorption	The equilibrium adsorption capacity (Q_e) mg/g	Removal %
	10	1.636 ±0.17	4.180 ±0.08	83.62 ±1.71
Ni (II)	25	5.973±0.34	9.516±0.17	76.10±1.38
	50	19.346±0.63	15.663±0.30	61.30±1.26
	10	1.993 ±0.22	4.000 ±0.11	80.06±2.20
Zn (II)	25	7. 156 ±0.15	8.923 ± 0.08	71.37± 0.62
	50	20.180±0.62	14.913 ±0.08	59.64± 0.62

Adsorption experiment

Nickel (Ni) salts can produce an increase of harmful effects on human health such as allergy and contact dermatitis *(Kasprzak et al., 2003)*. Zinc (Zn) is a critical element for life, but it is also highly toxic when used in high concentration. Adsorption properties of PVA/CNC magnetic composite for removal of the tested ions from aqueous solutions were investigated. Forty mg of PVA/CNC magnetic adsorbent were added to 20 ml of each of Ni (II) and Zn (II) solutions at different concentrations (10, 25 and 50 ppm) under continuous stirring for 30h at room temperature . Data presented in Table 1 indicate that the highest removal of Ni (II) and Zn (II) was reached at a concentration of 10 µg/ml with percentages of 83.62 % and 80.06 %, respectively. It is also observed that the removal percentage of a heavy metal is inversely correlated with the heavy metal concentration. Our findings agreed in a large extent with that obtained by *Li et al. (2011*) who declared that PVA/chitosan could separately remove Malachite green (MG) and Cu^{2+}. Additionally, (*Monier and Abdel-Latif,* 2012) developed a magnetic adsorbent based on phenylthiourea and chitosan as a proper treatment for wastewater containing harmful concentrations of heavy metals.

Fig.6 describes the adsorption efficacy of Ni (II) and Zn (II) by PVA/CNC magnetic composite which extensively increases with increasing contact time. At equilibrium (after 25h), adsorption capacities were 4.00 and 4.18 mg/g for Zn (II) and Ni (II), respectively (Table, 1). The adsorption route involved two stages: the first stage is rapid (till 15h); and the second is slow in which the adsorption equilibrium reached in 20h. In the rapid stage, surface of adsorption on PVA/CNC beads was not fully occupied with the heavy metal. The slowness of the second stage may be due to the diminution of the effective sites on the surface of the tested adsorbent. This finding agreed with that obtained by *Sureshkumar et al.* (2010) who found a rapid adsorption in the first 4h of contact period and a slow stage in which the adsorption process reached equilibrium in the third day when tripolyphosphate-chitosan composite was used in the treatment of uranium polluted solutions.
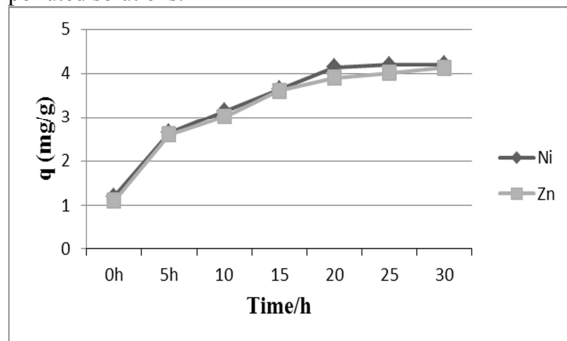

Fig. 6. Effect of contact time on Ni (II) and Zn (II) sorption

Generally, the highest adsorption capacities of Ni and Zn were attained at initial concentration of 50μg/ml with the values of 15.66 and 14.91 mg/g adsorbent, respectively (Table, 1). This result was moderately higher than that obtained by *Ul-Haq et al.* (2017) who stated that sorption capacity of Ni at initial concentration of 40μg/ml from aqueous solutions ranged between 8.567 mg/g to 9.165 mg/g when *pea peel* was used as a natural sorbent. So it could be suggested that the developed magnetic nanocrystal composite from *pea peels* in this study increases their ability to adsorb the heavy metal from aqueous solutions.

Morphological characters of PVA/CNC magnetic composite before and after adsorption process was examined by SEM (Fig.7 A, B and C). It is noticed that the prepared magnetic composite is smooth surface well-shaped spheres with about 1 mm diameter. The adsorbed heavy metals on the surface of magnetic beads were studied by EDX. The EDX spectra of PVA/CNC bead before and after Ni (II) and Zn (II) adsorption is presented in Fig 7 A, B and C. Micro pores of magnetic composite in Fig 7 B and C were filled by metal ions and significant decreases in the surface roughness were occurred and so the heavy metal loaded magnetic composite (Fig 7 B and C) seemed to be smoother than that of non-treated composite (Fig 7A). The smoothness of PVA/CNC magnetic composite after sorption clarified the occurrence of Ni (II) and Zn (II) in the composite after adsorption. The occurrence of heavy metals on the surface of chitosan/silica gel beads was previously proven using SEM-EDX (Gandhi and *Meenakshi, 2012)*.The interpretation of SEM-EDX and FTIR of PVA/cellulose magnetic composite before and after adsorption of Ni (II) and Zn (II) explained that -OH groups were involved in the adsorption of these metals.
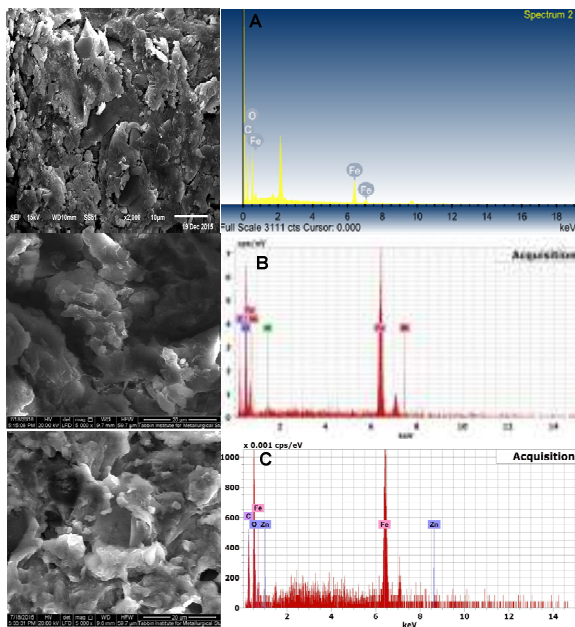

Fig. 7. The SEM-EDX spectra of PVA/cellulose nanocomposite before (A) and after (B), (C) Ni (II) and Zn (II) sorption.

CONCLUSION

Pea peels purified cellulose nano-crystals were used to prepare PVA/CNC magnetic composite which was entirely characterized and employed as an adsorbent for the removal of Ni (II) and Zn (II) from aqueous solutions. The maximum sorption capacities of the developed magnetic composite recorded were 15.66 and 14.91mg/g for Ni (II) and Zn (II) from aqueous solutions, respectively. It could be elucidated that PVA/CNC magnetic composite is a good resource and eco-friendly adsorbent for removal of harmful ions from aqueous solutions.

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تحضير تراكيب ذات خواص مغناطيسية مكونة من بلورات النانوسليلوز وكحول عديد الفينيل وكفاءة استخدامها فى التخلص من ايونات النيكل والزنك من المحاليل المائية

داود حسنى الحنفى و محمد عبد الحميد طاهر

قسم الكيمياء الزراعية – كلية الزراعة ـ جامعة المنصورة

ان المعادن الثقيلة عناصر وهناك مزيدا من الاهتمام فى الوقت الحالى للعمل نحو التخلص من مثل هذه الملوثات البيئية من المياة العادمة . وقد استخدمت مخلفات قشور البسلة الخضراء فى تنقية السليلوز الذى بنيت عليه هذه الدراسة. وقد استخدمت التراكيب المغناطيسية لكحول عديد الفينيل والنانو سليلوز المنقنوالمحضرة مسبقا فى دراسة تأثير زمن الاتصال على القدرة الادمصاصية نحو التخلص من ايونات النيكل والزنك . وقد اظهرت النتائج ان الكفاءة الادمصاصية للنيكل والزنك بواسطة هذه التراكيب المغناطيسية تزداد مع زيادة فترة الاتصال والتى تصل لمرحلة الاتزان بعد ٢٥ ساعة . ان القدرة الادمصاصية عند الاتزان كانت ٤٫١٩ و ٤٫٠٠ مجم/جم للنيكل والزنك على التوالى وقد كانت أعلى نسب مئوية لازالة المعادن هى ٨٣٫٨٦ و ٨٠٫١٣ % عند تركيز ١٠ ميكروجرام/مل من النيكل والخارصين على التوالى . وقد استخدمت تحليلات طيف الاشعة تحت الحمراء والميكرسكوب الالكترونى الماسح قبل وبعد العمليات الادمصاصية للنيكل والزنك بواسطة التراكيب المغناطيسية لكحول عديد الفينيل وبلورات النانوسليلوز وأظهرت ان مجاميع الهيدروكسيل الوظيفية تشارك بدور أساسى فى ادمصاص النيكل والزنك . وفى النهاية يمكن تلخيص الامر فى ان التراكيب المطورة من كحول عديد الفينيل وبلورات نانو السليلوز ذات الخصائص المغناطيسية يمكن اعتبارها مادة ادمصاص واعدة نحو التخلص من النيكل والزنك من المياة العادمة .