

Preparation, Characterization and Adsorption Evaluation of old Newspaper Fibres using Basket Reactor (Nickel Removal by Adsorption)

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ABSTRACT: In this work, old newspaper fibers (ONF) bleached with H₂O₂, treated with KOH and treated with NaOCl were investigated as potential adsorbents. The characterization of the produced fibers using FT-IR, SEM and particle size distribution have been carried out and tested for the removal of Ni (II) from aqueous solutions. The results indicated that the fibers treated with KOH give the highest %removal of Ni (II) with 88%. Two different reactor designs (batch and basket reactor) with different variables were studied. The results indicated that the equilibrium time was 30 min. and the removal of Ni (II) increased significantly as the pH increased from 2.0 to 6.0 and decreased at pH range of 6.5–8.0. The adsorption of Ni (II) onto ONF treated with KOH using batch and basket reactors follows the Langmuir isotherm. The pseudo second order kinetic model provided good correlation for the adsorption of Ni (II) onto ONF treated with KOH for both batch and basket reactors.

Key words: Old newspaper fibers, Heavy metals, Nickel, Adsorption, Wastewater

INTRODUCTION

Petrochemical industry is one of the most important industries that produce our daily life needs of chemical materials (Jafarzadeh 2008). Although petrochemical industries have several benefits for our life, yet they are considered as a real source of environmental pollutants. Nowadays the main focus is how to control the pollution and discharge from the petrochemical industries and has to be addressed as a major international challenge to protect the environment against its impacts (Ghinwa et al., 2009). Wastewaters delivered from petrochemical industries are characterized by large quantities of organic pollutants such as polycyclic and aromatic hydrocarbons, phenols, heavy metal derivatives, and other chemicals (Nocito et al., 2007). Some metals that have high level of toxicity especially Pb (II), Hg(II), Cd(II), Ni(II) and Cr(VI) are of great concern to our life. Their concentrations must be reduced to acceptable levels before discharging them into environment (Aslam et al., 2004). Toxicity levels of

heavy metals depend on the type of metal, its valence states, its biological role and volume or concentration (Michael Hogan et al., 2010; Weber et al., 1980). According to World Health Organization (WHO) the metals of most immediate concern are chromium, copper, zinc, iron, nickel, mercury and lead (World Health Organization 2010). Ni (II) containing wastewaters are common as it is used in a number of industries including electroplating, batteries manufacturing, mining, metal finishing and forging.. The higher concentration of Ni (II) in ingested water may cause serious lung and kidney problems aside from gastrointestinal distress, pulmonary fibrosis and skin dermatitis and it is known that nickel is human carcinogen (Borba et al., 2006). Removal of Ni (II) from aqueous medium are commonly accomplished by several well-known methods including ion exchange (Alyüz et al., 2009; Shaidan et al., 2012), precipitation (Papadopoulos et al., 2004; Blais et al., 2008),

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coagulation(De Mello Ferreira et al.,2013), ultrafiltration (Katsou et al.,2010) biosorption (Gabr et al., 2008), adsorption (Heidari et al., 2013), membrane filtration (Borbély et al., 2009).....etc.

Among all these techniques, adsorption has been found to be a promising method that does not have the disadvantages of the other method such as low efficiency and high cost (Samarghandi et al., 2011; Krishnan et al.,2011; Syed et al., 2014; Ali 2010, 2012, 2014 ; Ali et al., 2012, 2006).Nowadays, low cost, environmentally benign and abundant resources are getting widespread attention for Ni (II) effluent treatment, where agricultural waste and biomass products are converted into a value-added system as effective adsorbents(Bansal et al.,2009; Ossman et al.,2014; Cuhadaroglu et al.,2008). Lignocellulosic materials owing to their chemical compounds and complex structures of cellulose, hemicellulose and lignin can be used for Ni (II) adsorption either with slight or no modifications or treatment through physical and chemical means to have similar properties as activated carbons. Various lignocellulosic and agricultural wastes have been forwarded to maximize the adsorption capacity of Ni (II) from wastewaters (Garg et al., 2008; Shukla et al.,2005; Kumar et al.,2009). The aim of this work is to study the possibility of using old newspaper fibers (ONF) bleached with H_2O_2 , treated with KOH and treated with NaOCl as adsorbents for removal of Ni (II) from waste water using batch and basket reactors. Important parameters that affect the adsorption process such as; time, initial concentration of Ni(II), pH, and dosage of adsorbent were studied along with the adsorption isotherms and kinetics .

MATERIALS & METHODS

All chemicals used in the experiments are of analytical reagent grade. The Old newspapers were collected and cut into small pieces, treated with 1.2% w/w NaOH, 1.0% w/w H_2O_2 for removing foreign materials like grease, black ink, and act as bleaching material whereas the fibres were then washed several times with distilled water till the pH of the supernatant water layer of the pulp was around 6.5 to 7.0. Then the Old newspapers fibers (ONF) were shredded into small pieces by mechanical mixer. The produced (ONF) were treated chemically as follows, ONF fibers were soaked in 0.4% (wt%) NaOCl solution for 60 min. After this, the treated fibers were washed with distilled water and dried at 80 °C for 1.5 hrs.

Also,ONF fibers were soaked in 0.4% (wt%) KOH solution for 60 min .After this, the treated fibers were washed with distilled water and dried at 80 °C for 1.5 hrs.

The surface morphology of the bleached old newspaper fibers (ONF) and treated old newspaper fibers were analyzed using different magnifications of scanning electron microscopy (SEM, JEOL JSM 6360LA). The surface functional groups with binding sites and structure of the solid materials were studied by Fourier Transform Infrared Spectroscopy (FTIR-8400S, Shimadzu). The FT-IR spectra were recorded at a wave number between 500 /cm and 4000 /cm.

Fig.1 shows the experimental setup for the basket reactor used in the present work. It consisted of a 2L Plexiglass container. ONF were packed in four rotating perforated PVC baskets of 3 cm diameter, 6 cm height and 2 cm thickness. The four baskets were treated with Epoxy. The basket was mounted centrally on an isolated shaft connected to a controlled variable speed motor. The basket rotational speed was varied within the range of 50 to 100 rpm.

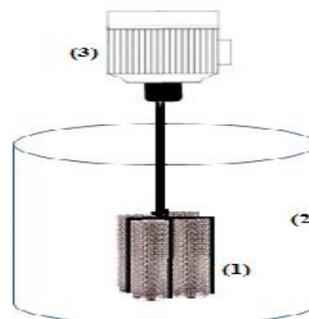


Fig. 1. The Basket Reactor. Rotating packed impeller (2) Plexiglass container (3) Motor

A stock solution of Ni (II) (1000 ppm) was prepared by dissolving 4.953 g of nickel nitrate, $Ni(NO_3)_2 \cdot 6 H_2O$ (analytical reagent grade) in 10 mL of concentrated sulphuric acid and diluted to 1 liter with de-ionized distilled water. Samples with various Ni (II) concentrations ranged from (25-100 ppm) were prepared from the stock solution. All experiments were carried out in batch reactor with magnetic stirrer and basket reactors, the amount of solute adsorbed per unit gram of adsorbent q_e (mg/g) was evaluated from the equation,

$$q_e = \frac{(C_0 - C_e) * V}{W} \quad (1)$$

Where C_0 is the initial heavy metal concentration (mg/l), C_e is the concentration of the solute in the bulk phase at equilibrium (mg/l), W is the adsorbent mass (g) and V is the solution volume (L).

RESULTS & DISCUSSION

Morphological analysis of ONF either bleached, treated with KOH or treated with NaOCl were performed

by scanning electron microscopy (SEM) in order to show the major features and morphology of the fibers. The morphological appearance is shown in Fig. 2, by increasing the magnification from 2000 to 10000 as shown in Fig. 2; it is observed that the surface contains high roughness and hollow cavities with porous structures.

Using an optical microscope, it was possible to observe that the ONF fibers have a grey color. The SEM of bleached ONF is shown in (Figs. 2 a and b). The treatment with KOH also preserves the grey color (Figs. 2 c and d). The chemical treatment with NaOCl also preserves the grey color (Figs. 2 e and f). As lignin was being removed during the treatment with KOH process (Pejic et al.,2009), the surface morphology of cellulosic materials became rough as the bonding between cellulose fibers had disintegrated as shown in (Figs.2 c and d). The treatment with NaOCl and KOH produced a scratch formation and a partial disintegration of the fiber, probably due to the removal

of the parts of the hemicellulose and lignin that interconnects the cellulose fibrils. The particle size distributions for the adsorbents are shown in Table 1. The data shows that the bleached ONF has mean particle size 162.4 μm while the treatment of ONF either with KOH or NaOCl reduced the particle size of the fibers and thus increased the surface area of the adsorbent which make it suitable for the adsorption process.

The peaks in the FT-IR spectrum of ONF prior to adsorption in Fig. 3 were assigned to various groups and bands in accordance with their respective wave numbers ($1/\text{cm}$) showing at the same time a complex nature of the adsorbent. The Treated ONF with either KOH or NaOCl showed absorption structures similar to bleached ONF but with different intensities. The broad band and intense peak around $3405/\text{cm}$ - $3346/\text{cm}$ for all ONF (treated and bleached) suggesting hydrogen-bonded ($-\text{OH}$) stretching vibration from the cellulose and lignin structure of the fibres (Brigida et

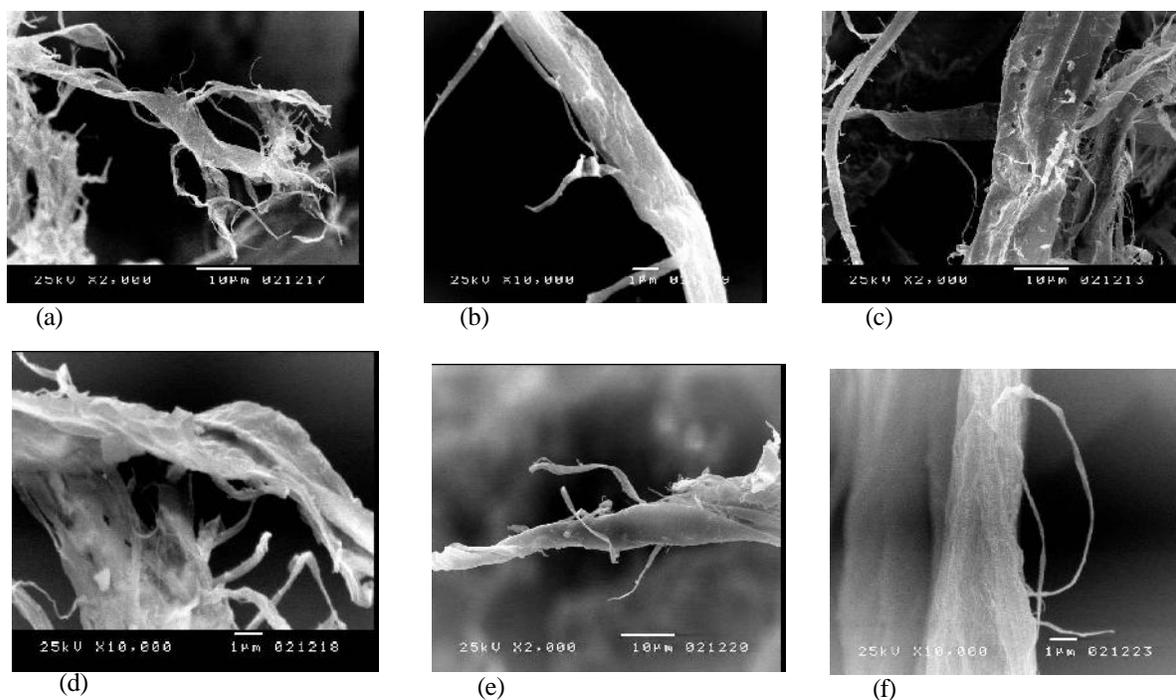


Fig. 2. SEM for old newspaper fibers at magnifications of 2000 and 10000 (a-b) bleached ONF (c-d) ONF treated with KOH (e-f) ONF treated with NaOCl

Table 1. Particle size distribution and mean diameter for the three adsorbents

adsorbent	Particle size distribution(μm)	Mean particle size (μm)
Bleached ONF	15.35-428.02	162.4
ONF treated with KOH	7.39-118.59	53.87
ONF treated with NaOCl	6.51-121.78	55.86

al.,2010).The bands have been shifted from 3346/cm for the bleached ONF to 3405 /cm for both treated ONF (with KOH or NaOCl),which is because of the change of cellulose crystal structure from cellulose (I) to cellulose (II) (Gwon et al.,2010).The peak at 2904 /cm - 2917 /cm is assigned to C–H asymmetrical stretching band in cellulose and hemicelluloses. At 1638 /cm , the absorption peak refers to aromatic skeletal vibration plus C=O stretching of hemicelluloses (Shukla et al.,2005).Peaks at 1428 /cm are indication for aromatic skeletal vibrations combined with –OCH3 in plane deformations (Nedjma et al.,2012).For 1373 /cm , the absorption peak refers to a symmetrical deformation of (C–H) in cellulose and hemicellulose groups for treated ONF with KOH or NaOCl (Brigida et al.,2010).The peak at 1059 /cm assigned as (C- O) stretching which starts to disappear when treating ONF with NaOCl .

Effect of contact time on adsorption of Ni(II) (100 ppm) using basket reactor equipped with the same dosage of bleached ONF, ONF treated with KOH or ONF treated with NaOCl (4g/l) is presented in Fig4. The % removal of Ni(II) were found to be 88%, 28% and 12% by using ONF treated with KOH, ONF treated with NaOCl and Bleached ONF respectively. According

to the results concluded from fig. 4 the ONF treated with KOH was used as the main adsorbent through the rest of the research. The adsorption of Ni (II) on (ONF) treated with KOH as adsorbent was studied as a function of contact time using batch reactor and basket reactor designs and the results are shown in Fig. 5 and Fig. 6 respectively. It was found that the rate of uptake of the Ni (II) is rapid in the beginning, where 78% and 88% adsorption is completed within 30 min for batch reactor and basket reactor designs respectively. Fig. 7 indicated that the Basket reactor equipped with ONF treated with KOH has higher % removal for Ni (II) than batch reactor equipped with the same amount of adsorbent.

The results of the experiments with varying adsorbent dosage are presented in Figure 8. It was found that with an increase in the adsorbent dosage from 1 to 4 g/L, the removal of Ni (II) increases from 72% to 85% in the batch reactor while in the basket reactor, the removal of Ni (II) increases from 18 % to 88%. These results are qualitatively in a good agreement with those found in the literature (Ming-qin et al.,2010).

The acidity of solution (pH) is one of the most important parameters controlling uptake of heavy

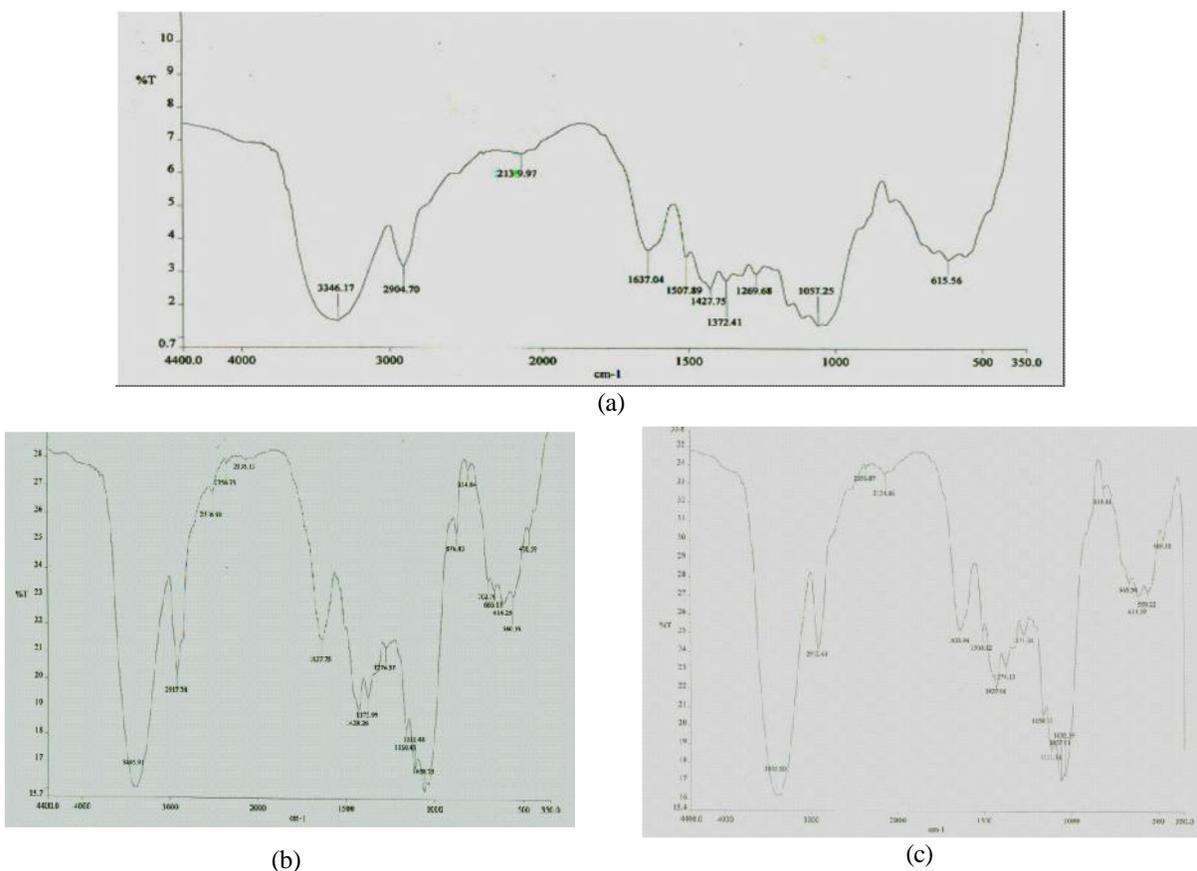


Fig. 3. FTIR for a) bleached ONF b) ONF treated with KOH c) Treated with NaOCl

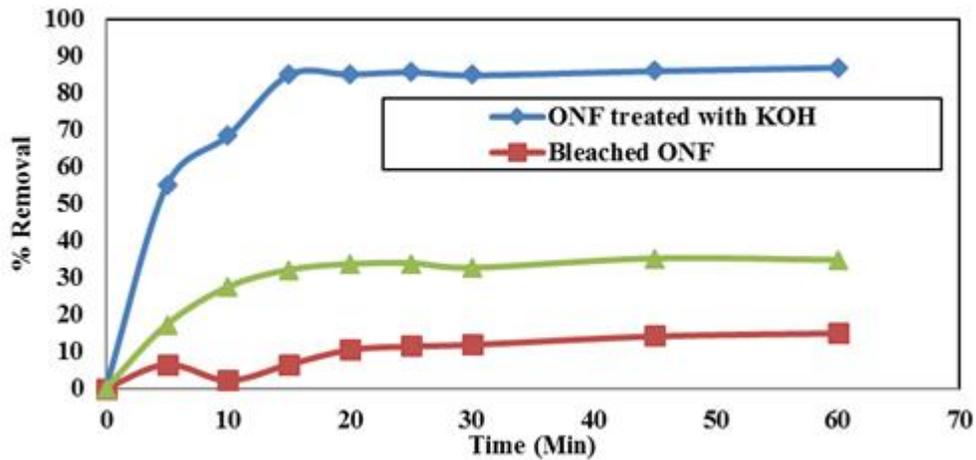


Fig. 4. Effect of contact time on the % removal of Ni by (ONF) treated with KOH, Bleached ONF and ONF treated with NaOCl. Conc. 100 ppm, pH is 6, adsorbent dosage is 4g/l and Temperature is 25 °C

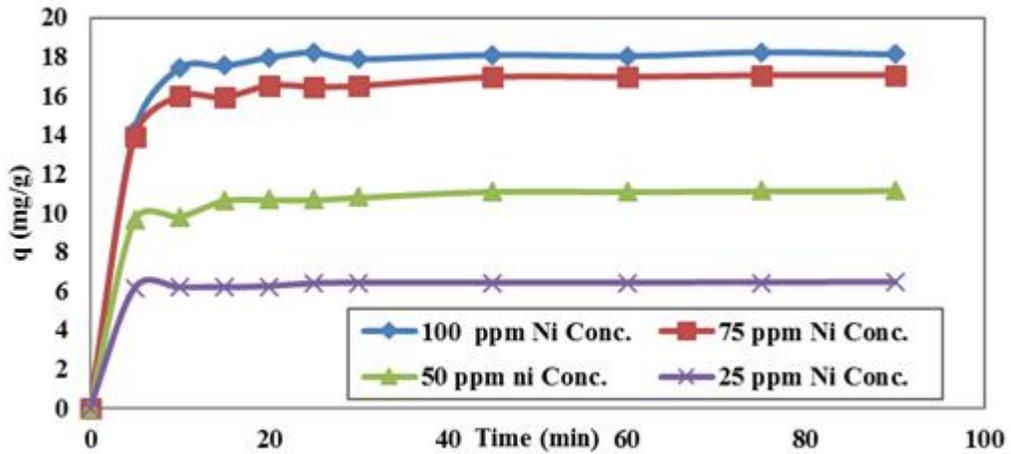


Fig.5. Effect of contact time on the adsorption capacity of ONF treated with KOH for Ni ions using batch reactor at different concentration and pH 6, adsorbent dosage 4g/l and Temperature is 25°C

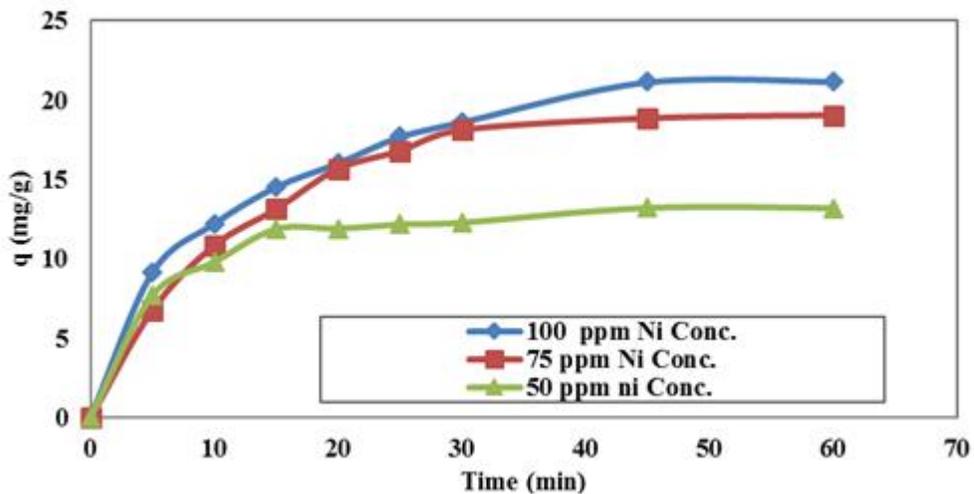


Fig. 6. Effect of contact time on the adsorption capacity of ONF treated with KOH for Ni ions using basket reactor at different concentration and pH 6, adsorbent dosage 4g/l and Temperature is 25°C

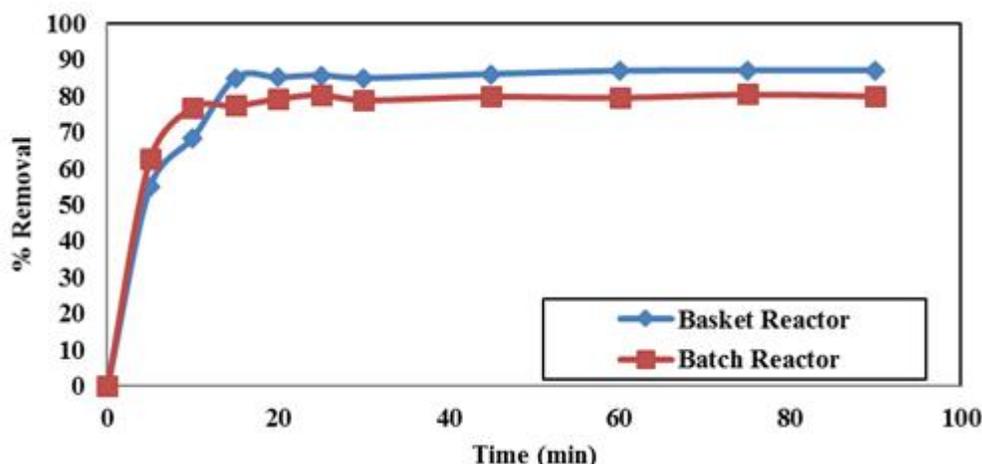


Fig. 7. Comparison between batch and basket reactor on the % removal of Ni (II) with ONF treated with KOH at 100 ppm concentration, pH 6, adsorbent dosage 4g/l and Temperature is 25°C

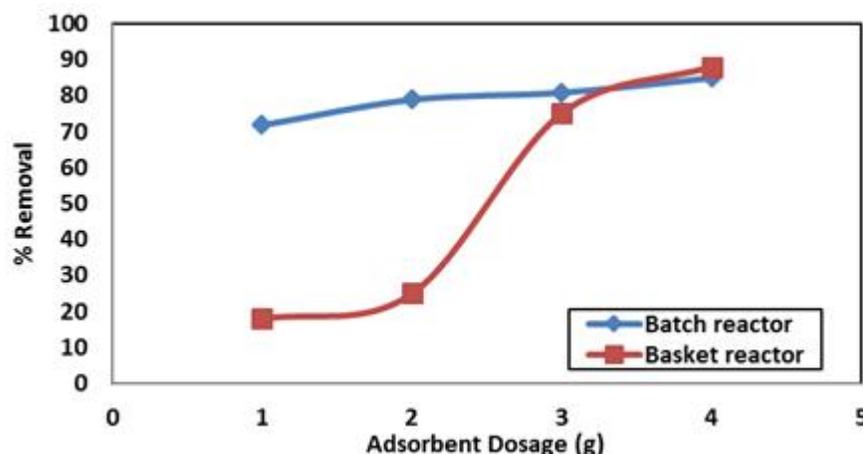


Fig.8. The effect of Adsorption dosage on % removal of Ni (II) by ONF treated with KOH using batch reactor at initial concentration of 100 ppm, pH 6 and Temperature is 25 °C using batch and basket reactors for 30 min

metals from aqueous solutions. In order to optimize the pH for maximum removal efficiency, experiments at 25 °C were carried out by taking into contact 100 mL of 100 ppm Ni (II) solution with 1g/l of ONF fibers treated with KOH and contact time (30 min) was kept constant by stirring and adjusting continuously the solution pH with HCl and NaOH solutions at selected pH of 2, 4, 6 and 8. Fig. 9 describes the effects of pH on adsorption of Ni (II). The adsorption of Ni (II) was found to be favored when the initial solution pH was between 2 and 6. With increasing the solution pH from 6 to 8, the adsorption efficiency decreased. However, a sharp decline in Ni (II) adsorption occurred when the pH was lower than 4.

The most widely accepted surface adsorption models for single-solute systems are the Langmuir and Freundlich models. The adsorption data was tested

using Langmuir isotherm equation in the linearized form,

$$\frac{C_e}{q_e} = \frac{1}{q_{max} b} + \frac{1}{q_{max}} C_e \quad (2)$$

Where C_e is the equilibrium concentration of the Ni (II) in solution ($mg L^{-1}$), q_e is the adsorption capacity at equilibrium ($mg g^{-1}$), b ($L mg^{-1}$) Langmuir constants, related to the binding constant and q_{max} ($mg g^{-1}$) is the maximum adsorption capacity. A plot of specific sorption (C_e/q_e) versus C_e gives a straight line of slope ($1/q_{max}$) and intercepts ($1/q_{max} b$) as given in Table 2 .

The Freundlich isotherm is commonly given by the following non-linear equation,

$$qe = K_F Ce^{1/n} \quad (3)$$

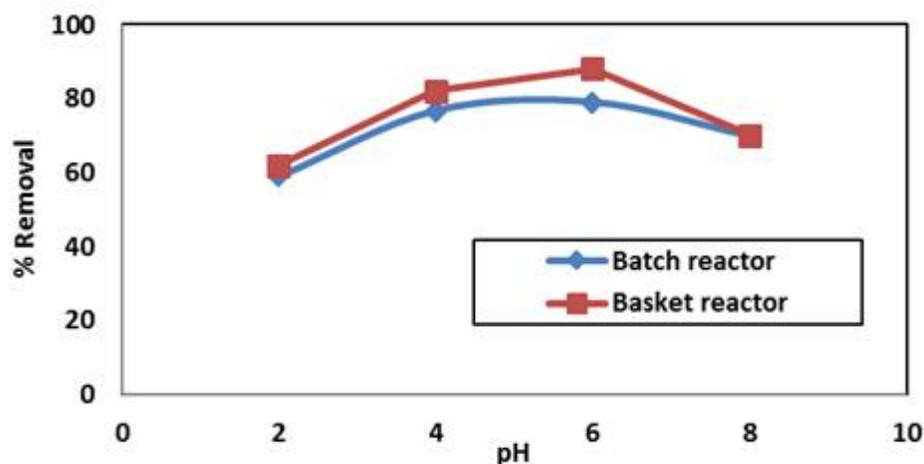


Fig. 9. The effect of pH on the % removal of Ni by ONF treated with KOH at initial concentration of 100 ppm and adsorbent dosage 4g/l and Temperature 25 °C using batch and basket reactors for 30 min

where K_F is a Freundlich constant, related to the bonding energy. K_F can be defined as the adsorption or distribution coefficient and represents the quantity of Ni(II) adsorbed onto adsorbent for unit equilibrium concentration. $1/n$ indicates the adsorption intensity of Ni(II) onto the sorbent with surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. Eq. (3) can be linearized according to the logarithmic Eq. (4) and the Freundlich constants can be determined,

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

The applicability of the Freundlich sorption isotherm was analyzed, using the same set of experimental data, by plotting $\log(q_e)$ versus $\log(C_e)$. The data obtained from the linear Freundlich isotherm plot for the adsorption of Ni(II) on treated ONF with KOH is presented in Table 2. The correlation coefficients reported in Table 2 showed strong positive evidence that the adsorption of Ni (II) onto ONF treated with KOH follows the Langmuir isotherm for both batch and basket reactors for 100ppm Ni (II) concentration. The Dubinin–Radushkevich(D–R)model was also applied to estimate the characteristic porosity of the adsorbent, the apparent energy of adsorption and the characteristics of adsorption on micropores rather than on a layer-by-layer adsorption (Dubinin et al.,1965; Radushkevich et al.,1949). The D–R model has commonly been applied in the following Eq. (5) and its linear form can be shown in Eq. (6)

$$q_e = Q_m \exp(-B\epsilon^2) \quad (5)$$

$$\ln q_e = \ln Q_m - B \epsilon^2 \quad (6)$$

Where B is a constant related to the adsorption energy ($\text{mol}^2 \text{J}^2$), Q_m the theoretical saturation capacity (mg g^{-1}), the Polanyi potential, calculated from Eq. (7).

$$\epsilon = RT \ln(1+1/C_e) \quad (7)$$

The slope of the plot of $\ln q_e$ versus ϵ^2 gives B ($\text{mol}^2 (\text{kJ}^2)^{-1}$) and the intercept yields the adsorption capacity, Q_m (mg g^{-1}). The mean free energy of adsorption, E (kJ mol^{-1}), defined as the free energy change when one mole of ion is transferred from infinity to the surface of the solid from the aqueous phase in the solution, was calculated from the B value using the following relations(Kundu et al.,1949),

$$E = \frac{1}{\sqrt{-2B}} \quad (8)$$

It can be used to estimate the type of adsorption. If the value is in the range 8-16 kJ mol^{-1} , then the adsorption type can be explained by ion exchange, and if $E < 8$, the adsorption type is physisorption (Bhakat et al.,2006).The value of E calculated using Eq. (8) is 1.29 kJ mol^{-1} for treated ONF with KOH. It shows that the type of adsorption involves in this study is physisorption (physical sorption) which usually takes place at low temperature (Inglezakis et al.,2006; Lagergren et al.,1898).

The adsorption kinetic data were described by the Lagergren pseudo-first-order model which the earliest known equation is describing the adsorption rate based on the adsorption capacity. The linear form equation is generally expressed as follows,

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (9)$$

In order to obtain the rate constants, the values of $\log(q_e - q_t)$ were linearly correlated with t by plot of $\log(q_e - q_t)$ versus t to give a linear relationship from which K_1 and predicted q_e can be determined from the slope and intercept of the plot, respectively.

The adsorption kinetic may be described by the pseudo-second order model. The linear equation is generally given as follows,

$$\frac{t}{q_t} = \frac{1}{K_2} \frac{1}{q_e^2} + \frac{1}{q_e} t \quad (10)$$

Table 2. Langmuir and Freundlich models for the removal of Ni(II) onto ONF treated with KOH using batch and basket reactors

Isotherm model	Batch reactor	Basket reactor
Langmuir		
Q^0 (mg g ⁻¹)	20.2	23.30
K_a (L mg ⁻¹)	0.51	0.475
No. of parameter estimated	2	2
Data point available	4	4
R^2	0.9825	0.9993
Freundlich		
1/n	0.3608	0.229
K_F (mg g ⁻¹)	7.296	10.834
No. of parameter estimated	2	2
Data point available	4	4
R^2	0.9014	0.9407

Table 3. Kinetic parameters for the adsorption of Ni (II) onto ONF treated with KOH for 100ppm Ni concentration

Conc. (ppm)	Pseudo first order kinetics			Pseudo second order kinetics		
	K_1 (min ⁻¹)	q_1 (mg g ⁻¹)	R^2	K_2 (g mg ⁻¹ min ⁻¹)	q_2 (mg g ⁻¹)	R^2
100 (batch reactor)	0.0633	2.767	0.511	0.081	18.31	0.9998
100 (basket reactor)	0.0749	20.942	0.9573	0.0039	25	0.9967
75 (batch reactor)	0.0352	3.7456	0.6352	0.0519	17.27	1
75 (basket reactor)	0.1007	22.775	0.9946	0.0041	22.988	0.9933
50 (batch reactor)	0.0331	2.562	0.6287	0.075	11.29	0.9999
50 (basket reactor)	0.0614	7.059	0.8973	0.018	14.144	0.9989
25 (batch reactor)	0.0376	0.741	0.5193	0.314	6.45	1

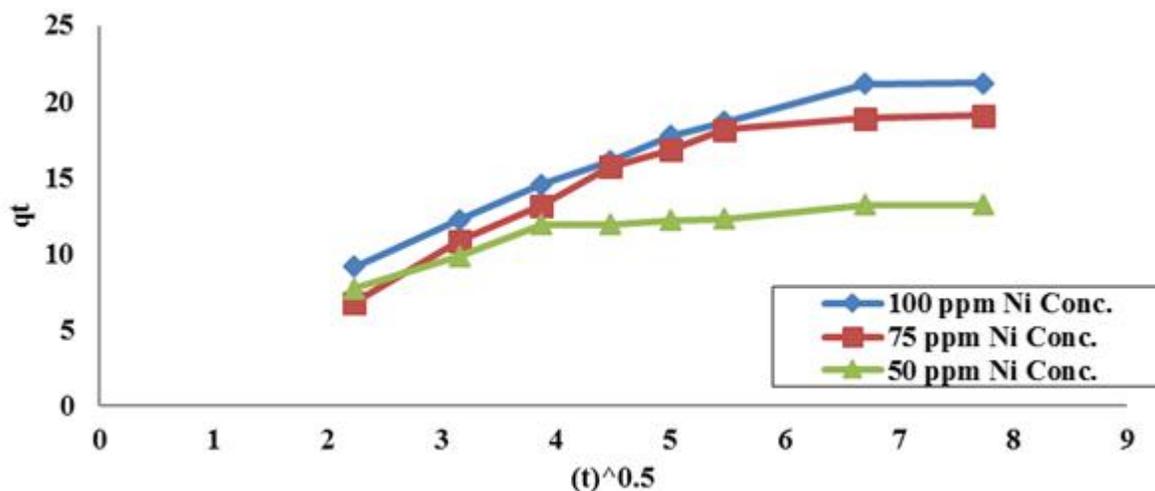


Fig. 10. Intra-particle diffusion for adsorption of Ni at different concentrations by ONF treated with KOH using basket reactor

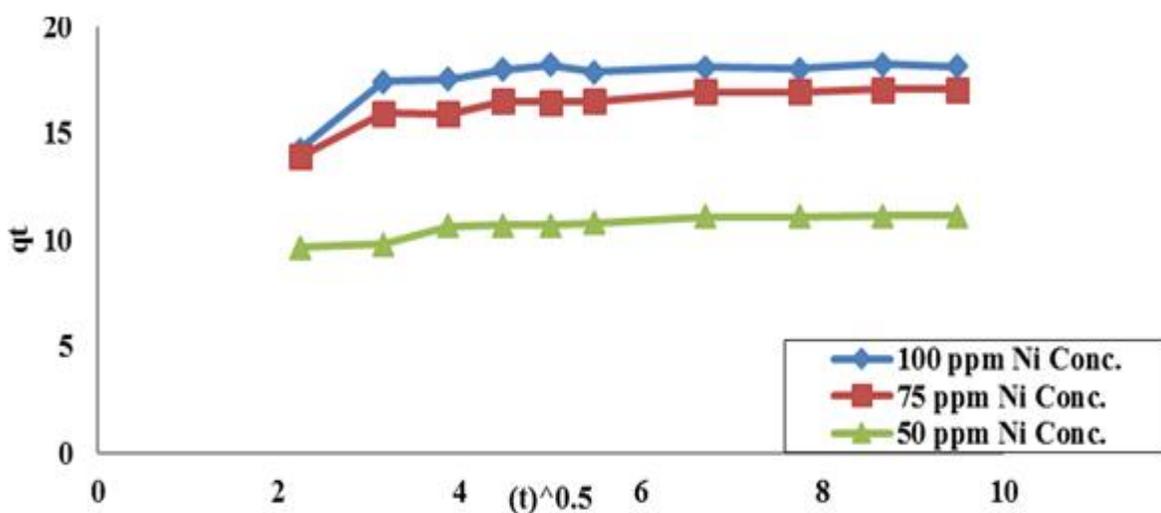


Fig. 11. Intra-particle diffusion for adsorption of Ni at different concentrations by ONF treated with KOH using batch reactor

If the second-order kinetics is applicable, then the plot of t/q_t versus t should show a linear relationship. Values of K_2 and equilibrium adsorption capacity q_e were calculated from the intercept and slope of the plots of t/q_t versus t . The linear plots of t/q_t versus t show good agreement between experimental and calculated q_e values (Table 3). The correlation coefficients for the pseudo second-order kinetic model are greater than 0.99, which led to believe that the pseudo second order kinetic model provided good

correlation for the adsorption of Ni (II) onto ONF treated with KOH.

The adsorbate transported from the bulk solution phase to the internal active sites occurs in several steps, where the rate of internal mass transfer is in most cases the rate-determining step in adsorption processes. Kinetic data were used further to check the possibility of intra-particle diffusion by using the Weber and Morris equation (Weber et al., 1963) expressed by the following equation;

$$qt = k_d t^{0.5} + c \quad (11)$$

where q_t (mg g^{-1}) is the amount of adsorbed Ni(II) at time t and k_d is the intra-particle diffusion rate constant ($\text{mg g}^{-1} \text{min}^{-0.5}$), and c is a constant determined from the intercept. If the plot of q_t versus $t^{0.5}$ gives a straight line passing through the origin, then the adsorption process is only controlled by intra-particle diffusion (Serpen et al., 2007). If multi-linearity in qt vs. $t^{1/2}$ plot is considered (that is, two or three steps are involved to follow the whole process) (Sun et al., 2003), fig. 10 and 11 show that the external surface adsorption occurs in the first step; the second step is the gradual adsorption step, where intraparticle diffusion is controlled; and the third step is the final equilibrium step, where the solute moves slowly from larger pores to micropores causing a slow adsorption rate.

CONCLUSIONS

Three cellulosic fiber materials have been prepared from old newspapers namely bleached ONF, ONF treated with NaOCl and ONF treated with KOH. The produced fibers were characterized and tested for removal of Ni(II) (100ppm). The % removal of Ni(II) found to be 88%, 28% and 12% by using ONF treated with KOH, ONF treated with NaOCl and Bleached ONF respectively. This indicated that the fibers treated with KOH give the highest % removal of Ni (II) as the results; It has been used as potential adsorbent for the removal of Ni (II) from aqueous solution. A new basket reactor consists of four poly vinyl chloride cylinder connected together and equipped with the adsorbent has been designed. The results indicated that the equilibrium time was 30 min. It was found that the removal of Ni(II) increased significantly as the pH increased from 2.0 to 6.0 and decreased at pH range of 6.5–8.0. The adsorption of Ni (II) onto ONF treated with KOH using batch and basket reactors follows the Langmuir isotherm. The pseudo second order kinetic model provided good correlation for the adsorption of Ni (II) onto ONF treated with KOH for both batch and basket reactors.

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