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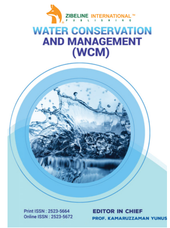

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RESEARCH ARTICLE

PREPARATION OF NOVEL HYBRID (ALMOND SHELL AND *PLEUROTUS SAJOR CAJU*) BIOSORBENT FOR THE REMOVAL OF HEAVY METALS (NICKEL AND LEAD) FROM WASTEWATER

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ABSTRACT

Level of contaminants (Nickel and Lead) in aquatic ecosystems has increased due to discharge of industrial effluents in water. Hence, there is a need to remove heavy metals (Nickel and Lead) from the water. For removing heavy metals from water, hybrid biosorbent (Almond shell and *Pleurotus sajor caju)* was prepared. To prepare a novel hybrid biosorbent (Almond shell and *Pleurotus sajor caju)* for the removal of nickel and lead from waste water the study was conducted in the department of chemistry, university of agriculture Faisalabad. The biomass was collected from local market of Chiniot. Hybrid matrix (Almond shell and *Pleurotus sajor caju)* and heavy metals (Nickel and Lead) were prepared. Waste water was interacted with the developed hybrid metals (Nickel and Lead) *and* hybrid bio sorbent (almond shell and *P.sajor caju)*.The maximum adsorption capacity q(mg/g) of nickel and lead obtained at l0mgL^{-l} concentration is in the following order; hybrid biosorbent(87)>*P.sajor caju(*65)> almond shell(54) and hybrid biosorbent(85)>*P.sajor caju*(57)>almond shell(45). The maximum uptake for nickel obtained by almond shell, *P.sajor caju*, hybrid biosorbent (56%), (66%), (90%) for lead and (47%), (61%), (89%) for nickel. The adsorption of nickel and lead follows the 2nd order kinetic model. FTIR spectra show that there are various functional groups, active sites present in hybrid biosorbent (Almond shell and *Pleurotus sajor caju)*. Maximum absorption of lead occurs at pH 5 and nickel at pH 3. The sorptions of heavy metals (Lead and Nickel) follow the pseudo 2nd order kinetic model. From the whole analysis it is concluded that Hybrid biosorbent calm of microbial and plant waste biomass was extremely functional in exclusion of lead and Nickel from wastewater.

KEYWORDS

biosorption, Nickel, lead, almond shell, *P.sajor caju*, hybrid biosorbent, Uv-visible spectrophotometer, FTIR.

1. INTRODUCTION

About two-thirds of the earth's surface occupied by water, water is obviously one of the main facets of human life (Hussaina et al., 2020). Water is the most essential element to life on earth. In its purest form it is odourless, colourless and tasteless. Level of contaminants in aquatic ecosystems has increased due to discharge of industrial effluents in water which in turn has led to water demand for domestic and industrial purpose (Abdi and Kazemi, 2015; Malik, 2020). Minamata disaster occurs in Japan because of pollution of heavy metals (Buasri et al., 2012). Water, comprising 70% of Earth's facade is undeniably the most valuable usual reserve existing on our globe. The living on the Earth would none exist without this invaluable solvent. In spite of it, pollution of water reserves is a frequent incidence. Hydrogen bonding and polarity are unique chemical properties by which water absorbs, adsorb and dissolve numerous compounds (Momodu and Anyakora, 2010). This valuable heritage is polluting by emerging contaminants including persistent organic matter, heavy metals, pesticides, herbicides, cynaotoxins, endocrine disrupting chemicals (EDCs) and by disposal of municipal, agriculture, industrial waste, excretion of pharmaceuticals (Tijani et al., 2013; La Farre et al., 2008).

The rapid growth of the mining, electrical engineering, and construction sectors has contributed to a significant issue of leakage of lead and nickel into the atmosphere (Huang et al., 2017). The non-biodegrading design causes lead to aggregate and moves into the food chains in the system. Also trace amounts of lead could also, pose risks for humans. Lead indicates liver, brain, nerve and sexual toxicity (Lai et al., 2016). Physical method, chemical method and biological approaches provide primary treatment of plumbed ions in wastewater (Huang et al., 2015). Bio-sorption has been a new area for the management of heavy metals in the past few years due to the high removal rate, low secondary contaminants and eco-friendly (Nguyen et al., 2013).

Significant quantities of toxic metals were created by metallurgical and industrial processes. Paper mills, battery plumbing and printed wiring unleash plumbing into our environment. Massive concentrations of human lead cause anaemia, brain injury and elevated blood pressure (Alam et al., 2012). Lead is harmful if its range is higher than 0.05 mg/dm. Bone cell plumage replace calcium, so both have the same load and scale. As Pb is

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low in blood, it decreases its conduction speed when its concentration in the blood increases, it causes nerve degeneration (Sanders et al., 2009). Nickel-cadmium batteries, pigments, fertilizers metal finishing, mining, metallurgical operations, tableware, plastic manufacturing, connecter and electroplating are the major source of nickel in our environment. Higher concentration of Ni in water causes vomiting, skin dramatis, diarrhea, nausea and pulmonary fibrosis. Nickel is cancer causing element.

Heavy metal ions can have been eliminated by several traditional techniques including chemical precipitation, reverse osmosis, electrochemical treatment techniques, ion exchange, membrane filtration coagulation, extraction irradiation and adsorption (Feng et al., 2012). Naturally occurring materials that are present in huge amount or some waste which are obtained from agriculture can be used as bio sorbents and these low-cost bio sorbents are environment friendly. Microbial cell uses as a bio sorbent for the removal of heavy metals is the replacement of traditional methods (Saifuddin and Raziah, 2007). Biosorption has a competency of biological materials to cumulate metals from waste water by metabolically acting or physical and chemical pathway of intake (Nasir et al., 2007). Biosorption has many advantages over conventional techniques because it does not produce chemical slime, more competent, easy to handle, it is highly particular and cheaper method for the removal of heavy metals from polluted water (Tunali et al., 2006).

While openly dangling biomass may have superior connection with adsorbents amide biosorption development, the dangling biomass is commonly not the sensible pattern form for the explicit value for the purification of waste water from heavy metals. Since cell restriction can increase its strength, and the calm of medication, it is widely used for the removal of heavy metals (Kanamarlapudi et al., 2018; Malik, 2020). Agricultural and microbial based materials have huge biosorption power as a bio sorbent, an innovative concept of generating a hybrid biosorbent were studied and an ingenious hybrid biosorbent matrix was produced by combining *pleurotus sajor-caju* and almond shell waste biomass. There have been previously no publications on removing heavy metals form the water. Therefore, we conducted a study of preparation of novel hybrid biosorbent for the removal of nickel and lead from waste water.

2. Methodology

To prepare a novel hybrid biosorbent for the removal of nickel and lead from waste water the study was conducted in the department of chemistry, university of agriculture Faisalabad. The biomass was collected from local market of Chiniot. hyphal suspension and Hybrid matrix were prepared and then immobilized. Heavy metals Nickle and lead was prepared, the effect of different experimental variable such as initial metal conc., pH, and contact time on absorption process was calculated in a batch mode. Waste water has been interacted with the developed almond shell, *P.sajor caju and* hybrid bio sorbent. Detection of nickel and lead has been done by using UV-visible spectrophotometer. The data which is obtained has been statistically analysed through simple linear regression. Flow chart diagram (figure 1) has shown the summary of the methodology which was performed to prepare a novel hybrid biosorbent for the removal of nickel and lead from waste water.

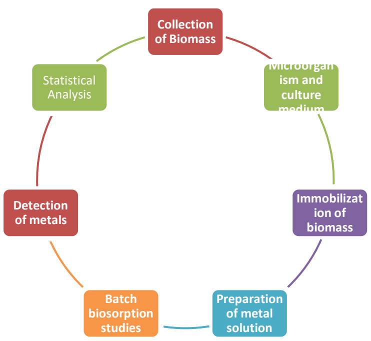

Figure 1: Flow chart diagram of the methodology

2.1 Collection of Biomass

The waste biomass of Almond was collected from local market of Chiniot. Water washed biomass was oven dried at 65 °C for 72 h. Dried biomass was cut and ground and then sieved

2.2 Microorganism and culture medium

For the preparation of hyphal suspension, the fungal strain of fungus *(Pleurotus sajor-caju)* was obtained by sub culturing on potato dextrose agar slants. Hybrid matrix was prepared according to the method described by (Ashraf et al., 2015).

2.3 Immobilization of biomass

Hybrid biosorbent matrix of *Pleurotus sajor-caju* and Almond shell was dissolved in (100 mL) of 2 % sodium alginate. The mixtures were homogenized. These mixtures then reacted with (0.1 M) $CaCl_2$ to obtain the proper-sized beads of biomass.

2.4 Preparation of metal solution

To prepare stock Ni and Pb solution (l000mg per liter) 3.80g of Ni $(NO_3)_2$ and l.60 g of $(PbNO_3)_2$ was dissolved in l00mL of deionized distilled water (DDW) and diluted quantitatively to l000mL using DDW. Different concentrations were prepared by adequate dilutions of stock solution with DDW.

2.5 Batch biosorption studies

The effect of different experimental variable such as initial metal conc., pH, and contact time on sorption process was calculated in a batch mode.

2.6 Detection of metals

Waste water has been interacted with the developed almond shell, *P.sajor caju and* hybrid bio sorbent. Detection of nickel and lead has been done by using UV-visible spectrophotometer.

2.7 Statistical Analysis

The data which is obtained has been statistically analysed through simple linear regression.

3. Results and Discussions

3.1 Influence of pH on metal ion biosorption

The pH is an important parameter in the adsorption process and particularly in the adsorption capacity. In order to evaluate the effect of initial pH on removal of nickel and lead from aqueous solution, batch adsorption experiment was performed at various pH values that ranges from 3 to 11 by keeping all other experimental conditions constant (50mg metals L^{-1}; agitation speed 220rpm; adsorbent material 0. l g; contact time l440min). The maximum adsorption capacity q obtained for nickel was (88) mg/g, achieved by hybrid bio sorbent at pH 3.0. There was a significant change in percentage removal of nickel in the range of pH of l.0-3.0, but beyond the pH 3.0 percentage removal decreased as pH increases. In case of lead the maximum adsorption capacities q(mg/g) (90) was achieved by hybrid bio sorbent at pH 5.0. There was a substantial change in percentage removal of lead in the range of pH of l.0-5.0, but beyond the pH 5.0 percentage removal decreased as pH increases.

The effect of pH for the adsorption of nickel and lead onto hybrid bio sorbent, *P.sajor caju,* almond shell, depicted in Fig 2. Adsorption capacities q(mg/g) of nickel and lead studied pH is as follows; almond shell (60), *P.sajor caju* (75) and hybrid biosorbent (88) and almond shell (66), *P.sajor caju* (74) and hybrid biosorbent (90), respectively. Most of the metal molecules exist in ionic form in the solution, and the solubility depends on the degree of dissociation. With increasing pH, the repulsive interactions decreased and the extent of Ni adsorption increased, presumably because of an ion-exchange mechanism between the surface protons and the Ni ions (Zhang and Wang, 2015). The adsorption level is significantly affected by surface charge of the adsorbent, which is highly dependent on the pH

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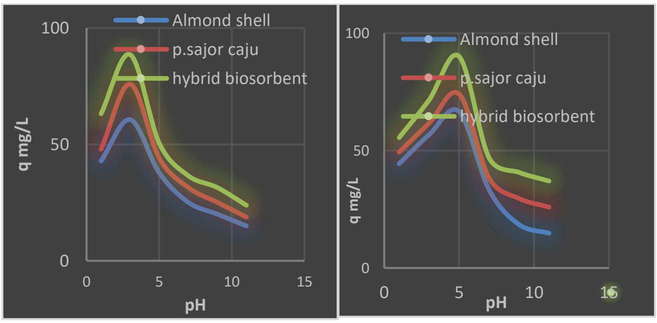
value of the solution (Attia et al., 2013). The pH 3.0 was selected optimum pH for adsorption of nickel onto hybrid biosorbent.

At lower pH, the biosorbent sites are concentrated by hydronium ions that constrain the approach of metal ions to cells, and on rising pH the hydroxyl groups reside on the binding site and exert a pull-on positive metal ion. At higher pH, metal hydroxide precipitation takes place, but sorption studies are unfeasible (Yang et al., 2015; Yu et al., 2017). Thus, at lower and higher pH, the sorption capacity is less, which is analogous to the results of earlier studies. As pH increases from 3–5, the percentage reduction of the Pb concentration increases because of the available binding sites, and at pH 6 the percentage removal decreases because of hydroxide precipitation. The maximum percentage removal of Pb (II) was 90% at pH 5 using hybrid bio sorbent.

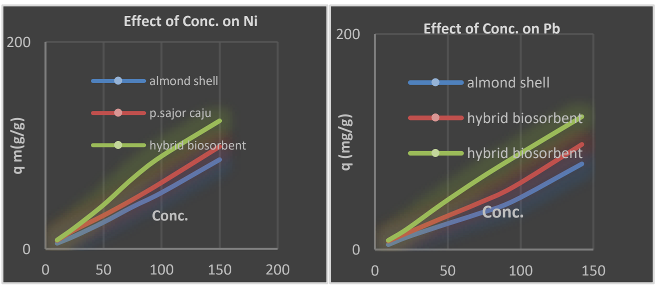


(a) (b)

Figure 2: Effect of pH on (a) Nickel and (b) lead

3.2 Effect of initial metal concentration

The concentration of metals is an important when studying the adsorption of metal in aqueous solution. The influence of metals concentration (l0 to 150ppm) on the removal of nickel and lead from the aqueous solution evaluated at pH 3.0 and 5.0 respectively with 0.l g adsorbent mass. The adsorption capacity q (mg/g) of almond shell, *P.sajor caju* and hybrid biosorbent for sequestration of metals under variable initial metal concentration 10, 25, 50, 75, 100 and 150 mgL^{-1}was evaluated (Figure 3). According to data revealed the uptake affinity q (mg/g) increase with initial metal concentration but percentage removal decreases with increasing metal concentration. The maximum adsorption capacity q(mg/g) of nickel and lead obtained at l0mgL^{-l} concentration is in the following order; hybrid biosorbent (87)>*P.sajor caju*(65)> almond shell(54)and hybrid biosorbent(85)>*P.sajor caju*(57)>almond shell(45) respectively. The rate of adsorption is a function of the initial concentration of the adsorbate. The removal of nickel and lead decreased with increment of the metal concentration. This can be explained by considering that, all adsorbents have a limited number of active sites and at a certain concentration, and the active sites become saturated. However, the adsorption capacity at equilibrium is increased with an increase in initial concentration. This is due to an increasing concentration gradient, which acts as a driving force to overcome the resistances to mass transfer of metal between the aqueous phase and the adsorbents (Pouretedal and Sadegh, 2014).



(a) **(b)**

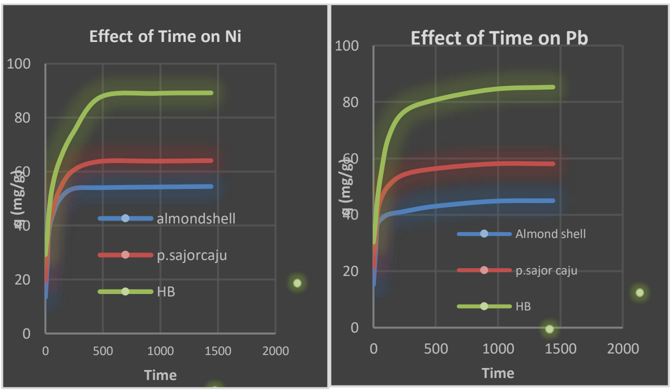
Figure 3: Effect of initial metal concentration on (a) Nickel and (b) lead

3.3 Effect of contact time

The effects of contact time on adsorption of both metals nickel and lead at 50 mgl^{-l} initial concentrations and 25^{0}C temperature were studied. The experimental data of this study is depicted in (Figure 4) It has been appeared that rapid initial uptake occurs and the adsorption capacity increases with concentration. The equilibrium achieved after having been shaking for less than 240 min. The maximum uptake for nickel obtained by almond shell, *P.sajor caju*, hybrid biosorbent (56%), (66%), (90%) for lead and (47%), (61%), (89%) for nickel respectively.

The contact time necessary to reach equilibrium depends on the initial metal concentration. The rate of adsorption on the surfaces would be proportional to a driving force times and area. As contact time increases, metals uptakes also increase initially. These changes in metal uptake may be due to the fact; at the initial stage, the higher driving force facilitates transfer of metal molecules to the surface of adsorbents and the availability of numerous active sites on the adsorbent. After that periods, the decrease in adsorption rate may be attributed to the reduction of numbers of remaining active adsorption sites and the long range diffusion effect of metal molecules (Meng, 2016).

Thus, a contact time of less than l20 min was invariably sufficient to reach equilibrium. The fast uptake of the metal molecules is due to solute transfer, as there are only sorbate and sorbent interactions with negligible interference from solute-solute interactions. The initial rate of adsorption was therefore great for high initial metals concentrations, the resistance to the metal uptake diminishes as the mass transfer driving force is increased (Antinoro et al., 2014).



(a) **(b)**

Figure 4: Effect of contact time on (a) nickel and (b) lead

3.4 Kinetic Modelling

Kinetic to metal adsorption governs the rate that determines the residence time and it is one of the most important characteristics defining the efficiency of an adsorbent. kinetic equation has been developed to explain the transport of metal onto various adsorbents (Ang et al., 2015). In order to investigate the kinetic mechanism of nickel and lead adsorption and potential rate controlling steps, two kinetic models pseudo-first-order and pseudo-second-order was fitted to the experimental data.

A comparison between pseudo-first-order kinetic model and pseudo-second-order kinetic models is depicted in (Table 1) The coefficient of correlation for the second order kinetic model was approximately equal to 1 and the estimated values of q_e mgg^{-l} agreed with the experimental ones shown in Figure 5 (a, b) and Figure 6 (a, b). Both facts suggest that the adsorption of nickel and lead follows the 2nd order kinetic model, which relies on the assumption that adsorption may be rate-limiting step. The initial concentration, equilibrium concentration and contact time were the experiment variables during contact time study (Konda, 2014).

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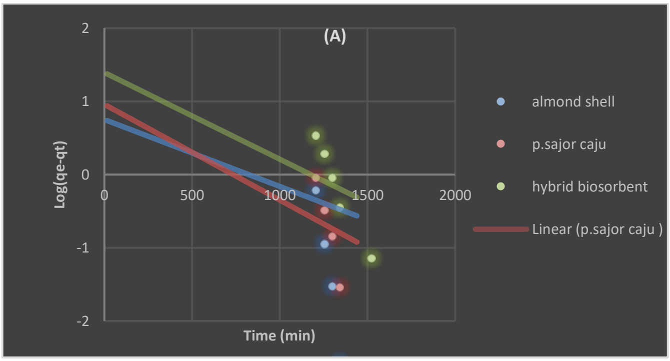

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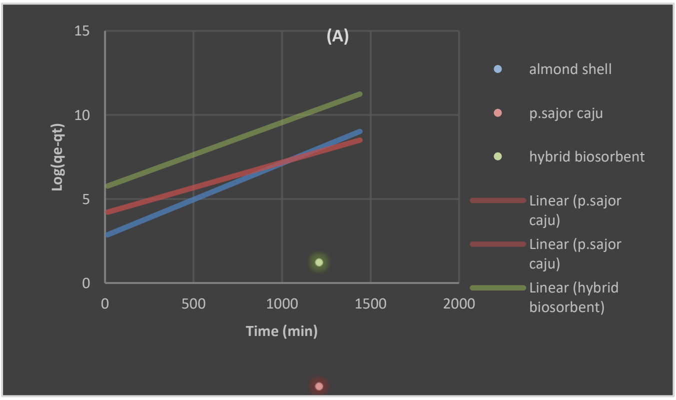
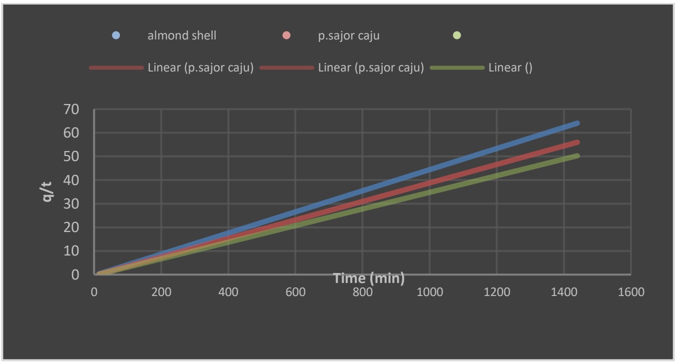


(b)

Figure 5: Pseudo I[st] order plot for (a) nickel and (b) lead



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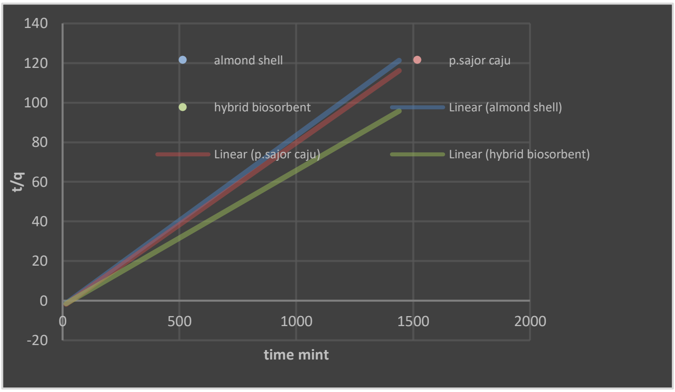


(b)

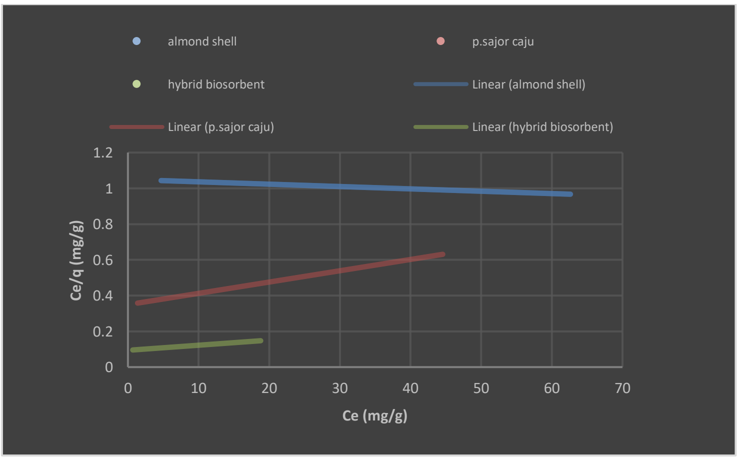
Figure 6: Pseudo 2[nd]order plot for (a) Nickel and (b) Lead

Table 1: Comparison between lagergren pseudo-first order and 2nd-order kinetic models for the adsorption of nickel and lead.

Metals	Biomass	Pseudo first order			Pseudo second order			
		q_e (mg/g)	K_{1ad} (min^{-1})	R^2	q_{exp} (mg/g)	q_e (mg/g)	K_{2ad} (mg/g min)	R^2
Nickel	**Almond shell**	7.749	0.00044	0.398	54.491	54.945	0.002127	0.9999
	P.sajor cjau	11.703	0.00068	0.473	64.073	64.516	0.001316	0.9998
	Hybrid biosorbent	28.464	0.00051	0.7007	89.176	90.909	0.000528	0.9996
Lead	**Almond shell**	8.429	0.00044	0.609	45.002	45.248	0.002654	0.9999
	P.sajor caju	12.305	0.00031	0.642	58.075	58.479	0.001757	0.9999
	Hybrid biosorbent	26.964	0.00051	0.7993	85.282	86.206	0.00065	0.9998

3.5 Equilibrium Modelling

Analysis of the experimental results by equilibrium adsorption equation often provides some insights into the sorption mechanism and the properties of the adsorbent. Several isotherm models describe the equilibrium between adsorbed metals ions on the cell and un adsorbed component remaining in the solution at certain concentration. A number of theories have been used as attempt to understand the adsorption process. The chemistry of adsorption is relatively complicated. To examine the relationship between adsorbed and aqueous concentrations at equilibrium, sorption isotherm models are wide1y employed for fitting the data, of which the Langmuir and Freundlich adsorption isotherm are the mostly used (Lalhruaitluanga et al., 2011).



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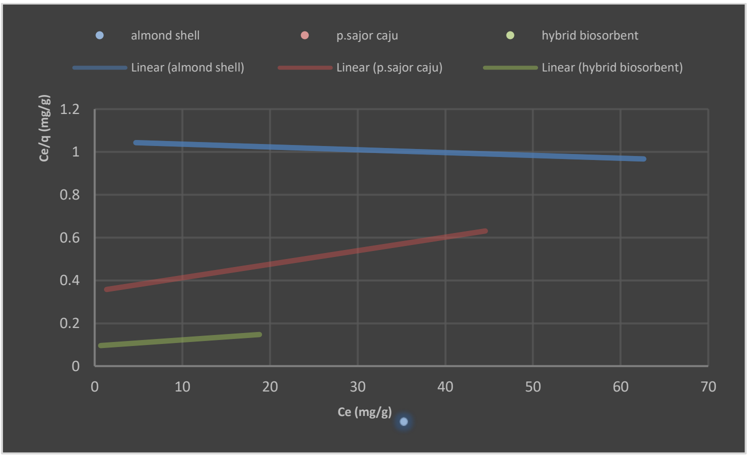
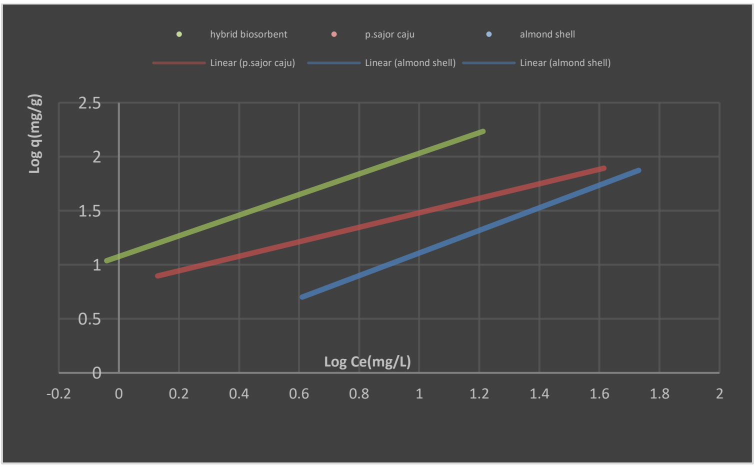


(b)

Figure 7: Langmuir isotherm plot on (a) Nickel and (b) Lead



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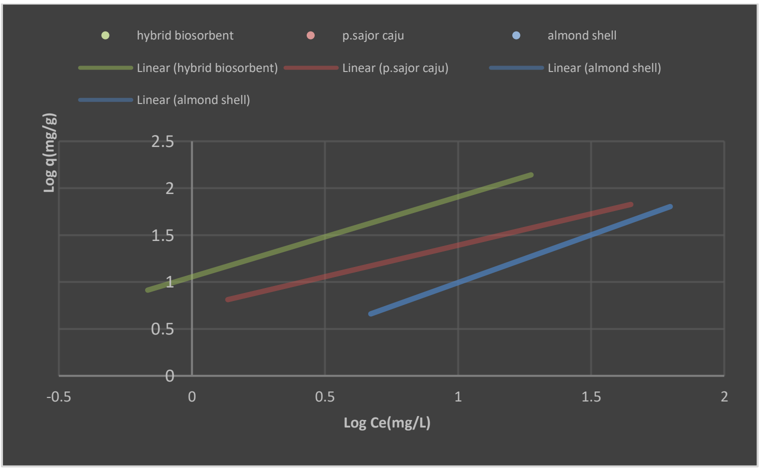

(b)

Figure 8. Freundlich isotherm plot on (a) Nickel and (b) Lead

Table 2: Comparison of Langmuir and Freundlich isotherm parameters of nickel and lead Characterization

Metals	adsorbent	Langmuir model			Experimental Value q(mg/g)	Freundlich model			
		Xm (mg/g)	K_1 (1/mg)	R^2		q_{max} (mg/g)	1/n	K_F (mg/g)	R^2
Nickel	**Almond shell**	416.66	0.0024	0.4333	86.264	84.80	1.0465	1.155	0.9984
	P.sajor caju	138.88	0.0072	0.6343	98.855	95.30	0.6708	6.462	0.9996
	Hybrid biosorbent	384.61	0.0026	0.2976	123.761	131.68	0.9548	11.939	0.9996
Lead	**Almond shell**	769.23	0.00124	0.052	79.513	70.71	1.0167	1.053	0.9916
	P.sajor caju	476.19	0.00522	0.1586	97.587	77.128	0.67	5.273	0.9338
	Hybrid biosorbent	357.14	0.030	0.3966	123.355	138.69	0.853	11.357	0.9495

3.5.1 FTIR spectra

Fourier-transform infrared spectroscopy (FTIR) is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-spectral-resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time. The term Fourier-transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum. Hybrid biosorbent has been prepared from *Pleurotus sajor caju* and almond shell. FTIR spectra show that there are various functional groups, active sites present in hybrid biosorbent, almond shell and *Pleurotus sjaor caju*. The FTIR spectroscopy is an important analytical technique which detects the chemical functional group in a molecule by its vibration characteristics. Hybrid biosorbent were adequately studied in the range of 500-45000cm^{-1} (Coates, J. 2006). Fig.9, 10 and 11 represent the spectra of almond shell, *Pleurotous sajor caju* and hybrid biosorbent.

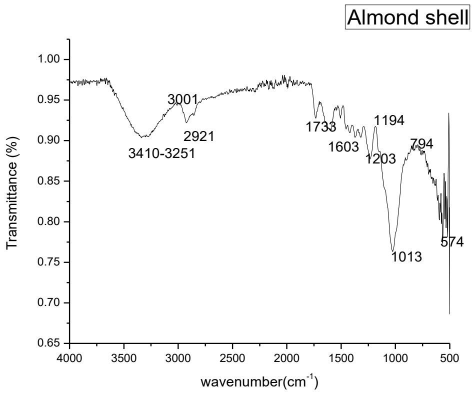

Figure 9: FTIR spectra of Almond shell

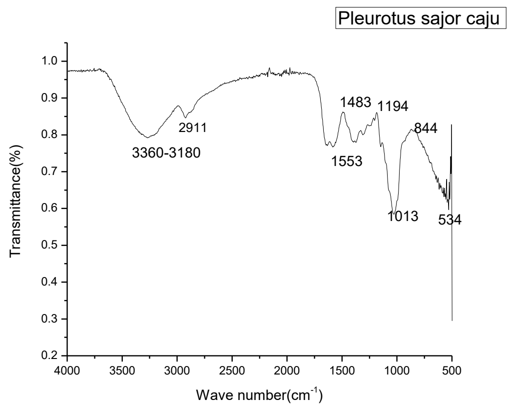

Figure 10: FTIR spectra of *P.sajor caju*

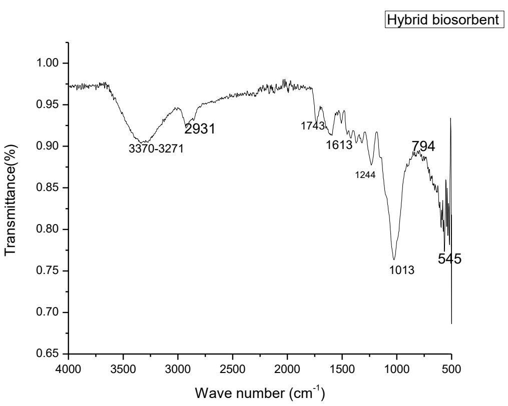

Figure 11: FTIR spectra of hybrid biosorbent

4. Conclusion

This study was conducted to prepare a novel hybrid biosorbent for the removal of nickel and lead from waste water. Our study reveals that capacities q(mg/g) of nickel and lead pH was almond shell (60), *P.sajor caju*(75) and hybrid biosorbent (88) and almond shell (66), *P.sajor caju* (74) and hybrid biosorbent (90). Our study revealed the uptake affinity q (mg/g) increase with initial metal concentration but percentage removal decreases with increasing metal concentration. The maximum adsorption capacity q(mg/g) of nickel and lead obtained at l0mgL^{-l} concentration is in the following order; hybrid biosorbent(87)>*P.sajor caju(*65)> almond shell(54)and hybrid biosorbent(85)>*P.sajor caju*(57)>almond shell(45). The maximum uptake for nickel obtained by almond shell, *P.sajor caju*, hybrid biosorbent (56%), (66%), (90%) for lead and (47%), (61%), (89%) for nickel. The adsorption of nickel and lead follows the 2nd order kinetic model. FTIR spectra show that there are various functional groups, active sites present in hybrid biosorbent, almond shell and *Pleurotus sjaor caju*. Maximum absorption of lead occurs at pH 5 and nickel at pH 3. The sorptions of lead and Nickel follow the pseudo 2nd order kinetic model. The Langmuir sorption isotherm model suited good to the Pb and Ni concentration records. From the whole analysis it is concluded that Hybrid biosorbent calm of microbial and plant waste biomass was extremely functional in exclusion of lead and Nickel from wastewater.

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