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Utilization of mesquite (*Prosopis juliflora*) wood biochar for adsorption of nickel ions in aqueous solution

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Abstract
In this adsorption study, removal of nickel from aqueous solutions has been inquired by mesquite (*Prosopis juliflora*) wood biochar as a low cost adsorbent. The rate of nickel adsorbed significantly increased with increase in initial concentration of nickel solution and adsorption rate gradually decreased as the dosage of biochar increased at the pH 6 and pH 7. The maximum adsorption rate was obtained 3.05 mg g^{-1} at the pH 7 with the lower adsorption dosage (0.25%) of biochar at 20 mg L^{-1}. The percent removal decreased with increased initial concentration of nickel and the percent removal increased with increased dosage of biochar at pH 6 and 7. The maximum percent removal was obtained 91.72% at the pH 7 at 1.0% biochar. Also, it helps in reduction of waste generation. Thus, the use of low cost adsorbents may contribute to the sustainability of the surrounding environment.

Keywords: Agricultural waste, Biochar, Heavy metal, Adsorption

Introduction
Heavy metal pollution is mostly spread in to the environment due to rapid industrialization and global urbanization (Kamari *et al.,* 2014) [1]. Heavy metals are non-biodegradable, bio accumulate and highly toxic in terrestrial and aquatic living organisms (Ghani *et al.,* 2013 and Yuvaraja *et al.,* 2014) [4, 22]. Nickel is one crucial heavy metal entering water sources through industrial activities like electroplating, batteries and metallurgical industries (Wantala *et al.,* 2014) [20]. The concentration of nickel in Coimbatore sewers ranging from 9.0-16 mg L^{-1} (Pavitharapriya *et al.,* 2015) [13]. In India is estimated that about 38,254.0 million litres day^{-1} (MLD) of wastewater is generated in urban centre (Kaur *et al.,* 2013) [8]. From this 1077.21 MLD of waste water are produced in Tamil Nadu. The alarming health issues of skin allergies due to nickel in wastewaters have increased the need to develop remediation measures to remove metals from water matrices (Ucar *et al.,* 2014) [19]. There are different methods of treatment available to remove heavy metals in wastewater such as adsorption, membrane filtration, chemical precipitation, ion exchange, flocculation, extraction and coagulation (Li *et al.,* 2014 and Tasar *et al.,* 2014) [10, 17].

Biochar, a pyrolysed carbon rich-porous material from various organic waste materials (Inyang *et al.,* 2016) [6]. Biochar can be used as a low-cost substitute for activated carbon used in the presence of pollutants (Cernansky, 2015) [2]. Furthermore, biochars are considered as a universal sorbents in the removal of organic and inorganic contaminants in both soil and water (Ahmad *et al.,* 2014) [1]. Additionally, biochars with high sorption capacity are considered as suitable feed stocks. If pyrolysed, it opens up a variety of functional groups on surface of then making it suitable to stop variety of contaminants. (Doumer *et al.,* 2016) [3]. In this study, the potential of using Mesquite wood biochar as a low cost biosorbent for removal of nickel in waste water.

2. Materials and Methodology
2.1 Adsorbent
Mesquite (*Prosopis juliflora*) wood was collected from Paramakudi in Ramanathapuram district. The collected samples were air dried at room temperature for 2-3 days then converted into biochar through pyrolysis at 450˚ C. The biochar samples were ground and sieved to 2.0 mm particle size.

Then the *Prosopis* wood biochar (PWB) was kept in oven at 60° C for 48 hr to reduce the moisture content. After air dried *Prosopis* wood biochar stored in air tight container for further analysis.

2.2 Adsorbate

Stock solutions of nickel were prepared by dissolving 4.08g of nickel chloride ($NiCl_2$) in 1000 ml of 1.0% nitric acid solution. 1.0% HNO_3 was used to prevent hydrolysis of metal ions solution. Initial concentrations of metal ions like 5, 10, 15 and 20 mg L^{-1} were prepared by diluting the 100 mg L^{-1} of solutions. The pH of the metal solution was adjusted using 1.0M HCl and 1.0M NaOH solutions (Onundi *et al.,* 2010) [12].

2.3. Adsorption characterization of biochar

2.3.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (M/s. FEI - Quanta 250, Czech Republic) was used to record the surface texture and morphological characteristics of the biochar samples (Ucar *et al.,* 2014) [19]. The biochar samples were spreaded on double sided conductive carbon tap fixed on the stub. After attaining high vacuum the filament was turned on and adjusted to various required parameters like electron beam, intensity, spot size, voltage, emission current then the SEM images were captured and pore space was measured to study the adsorption properties of biochar.

2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

In this FTIR analysis, biochars were specially used to determine the functional groups present for each temperature and biomass, especially carbons. FT-IR spectra were recorded with 1.0 mg of biochar sample embedded in potassium bromide 30.0 mg (KBr) and then pressed into pellets separately and observed in a FTIR (Model 8400S of Shimadzu, Japan) using Attenuated Total Reflectance (ATR) technique having wavelength source 400-4000 cm^{-1} (Trakal *et al.,* 2014) [18].

2.4. Batch experiment study

Batch experiments were conducted using varied concentrations (0, 5, 10, 15, 20 mg L^{-1}) of nickel solutions. Different dosage of biochar (0.25, 0.5 and 1.0%) was added to each conical flask. To this 50 ml of nickel at different concentrations of 0, 5, 10, 15, 20 mg L^{-1} was added and shaken for 8.0 hrs at 250 rpm in mechanical shaker. After sorption, the solution was filtered using Whatman No.42 filter paper. The filtrate was analyzed using Flame Atomic Absorption Spectroscopy (AAS). The amount of heavy metal adsorbed per unit mass of the adsorbent (q_e) and the percent adsorption of the metal was calculated as follows equation 1.0 and 2.0.

$$q_e = (C_o - C_e)\, V/M \quad (1)$$

$$\% = [(C_o - C_e / C_o)] \times 100 \quad (2)$$

Where, C_o = Initial concentrations of the metal ion in solution (mg L^{-1}) C_e = Final concentrations of metal ion solution (mg L^{-1}) V = Solution volume (L) and M = Mass of the sorbent (g) (Onundi *et al.,* 2010) [2].

3. Result and Discussion

3.1 Scanning Electron Microscope of *Prosopis* wood biochar

The Scanning Electron Microscope (SEM) shows morphology of the adsorbent before adsorbed state (Fig 1). SEM Micrographs of porous structured *Prosopis* wood biochar in an regular oval shaped form. These SEM images were obtained at 4000x magnification with a pore size varying from 1.29 µm to 1.70 µm width and length was 4.69 µm to 8.51 µm. Thus, the pore space and agglomeration of biochar particles could enhance metal uptake and encourage mass transport of heavy metals through the inside of the sorbent which satisfies the results with Zhang *et al*. (2009) [23].

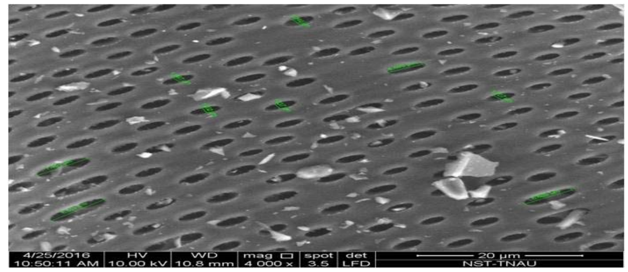

Fig 1: Scanning Electron Microscope of *Prosopis* wood biochar

3.2. Functional groups of *Prosopis* wood biochar

As a raw material, *Prosopis* wood is made up of polymers of lignocellulose (mainly hemicelluloses, cellulose, and lignin) after biochar having surface active groups (hydroxyl, carboxyl and amine *etc*) in their structures that are capable to bind heavy metals FTIR studies are used for prediction of different types of functional groups present in the adsorbent material. FTIR spectroscopy revealed that the result of PWB in Table 1. The peak as observed at 873.59 cm^{-1} is medium CH bonding of aldehyde functional group. A strong and medium intensity peak at 1267.00 and 1436.71 cm^{-1} is due to the alcoholic OH deformation mode. The medium peak at 1590.99 and 2352.73 cm^{-1} represents the deformation and stretching due to the presence of NH amine bonding functional groups. Similar CH, OH, NH bond were found for sunflower seed husks studied by Soldatkina *et al*. (2009) [16].

Table 1: FTIR spectroscopy of *Prosopis* wood biochar and their possible assignment

Functional Groups	Frequency (cm^{-1})	Bond	Intensity	Mode
Aldehyde	873.6	CH	Meadium	Deformation
Alcohols	1267.0	OH	Strong	Deformation
Alcohols	1436.71	OH	Meadium	Deformation
Amine Salts	1590.99	NH_2	Medium	Deformation
Amine Salts	2352.73	NH	Medium	Stretching

3.3. Effect of initial concentration of metal ions

3.3.1 Rate of adsorption of nickel with *Prosopis* wood biochar at pH 6 and pH 7

The rate of nickel adsorbed significantly increased with increase in initial concentration of nickel solution and adsorption rate gradually decreased as the dosage of biochar increased at the pH 6 and pH 7. The maximum adsorption rate was obtained 3.05 mg g^{-1} at the pH 7 with the lower adsorption dosage (0.25%) of biochar at 20 mg L^{-1} (Fig 2). Kavya (2016) [9] reported the rate of nickel adsorption of Coir waste biochar was 3.20 mg g^{-1} and adsorption rate gradually decreased with an increase the dosage of biochar. This scenario can be explained by the fact that there was a high probability of collision between adsorbent surface and metal ions at high concentrations. Therefore, the rate of diffusion of metal ions towards the adsorbent surface was expected to increase (Kamari *et al.,* 2014) [7].

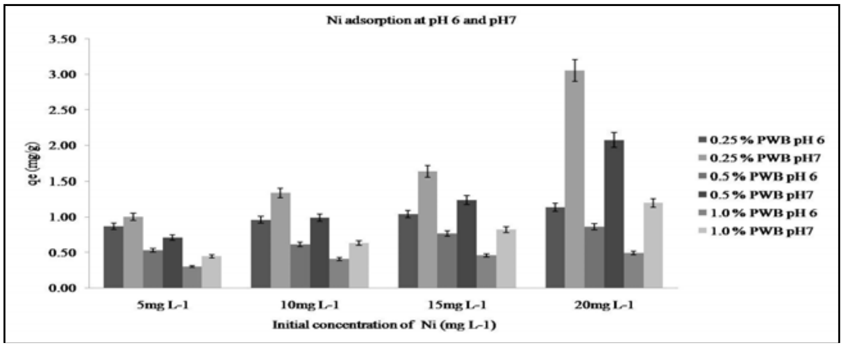

Fig 2: Effect of initial concentration on sorption rate of metal ions at pH 6 and pH 7 (mg g^{-1})

3.3.2 Percent removal of nickel with *Prosopis* wood biochar at pH 6 and pH 7

The percent removal decreased with increased initial concentration of nickel and the percent removal increased with increased dosage of biochar at pH 6.0 and 7.0. The maximum percent removal was obtained 91.72% at the pH 7.0 in the lower concentration of 5 mg L^{-1} at 1.0% dosage of biochar (Fig 3). Santuraki and Barminas (2015) [14] and Kavya (2016) [9] reported the nickel removal efficiency of coir waste biochar was 82.64% and the percent removal decreased with an increase in the initial metal ion concentration. This was due to low ratio at active sites available on the adsorbent surface to metal ions at high concentration (Yargic *et al.*, 2015) [21].

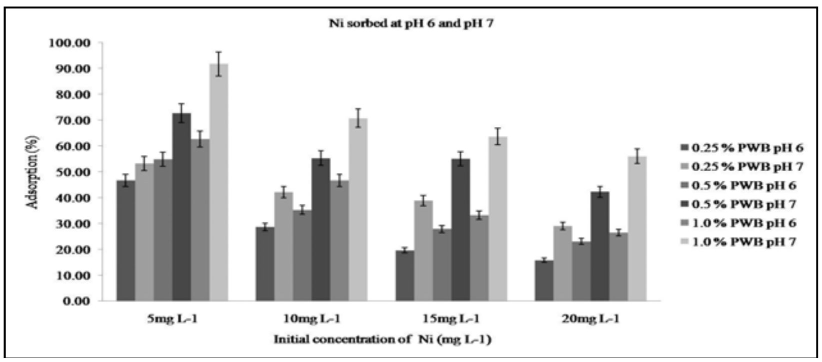

Fig 3: Effect of initial concentration on removal percent of metal ions at pH 6 and pH 7 (%)

3.4 Effect of adsorbent dosage

Adsorbent dosage is other important parameters that should be considered in contriving an effective adsorption system (Tasar *et al.*, 2014) [17]. The amount of adsorbent dosage is directly proportional towards the availability of the active binding sites (Yuvaraja *et al.*, 2014) [22] and the operational cost. The percentages of nickel removal increased significantly from 15.78 to 62.65% at pH 6.0 and from 28.96 to 91.72% at pH 7.0 for higher concentration to lower concentration of metal solution with the increase of the biochar dosage from 0.25 to 1.0%. The adsorption removal for nickel decreased drastically as the adsorbent dosage increased from 0.25 to 1.0% for increase the higher concentration of metal solutions (Fig 4). A similar observation was reported by Yargic *et al.* (2015) [21] for metal removal using tomato waste. This phenomenon can be explained by the fact that more active sites are available for metal ion binding at high dosage (Gupta *et al.*, 2013) [5]. An extra amount of adsorbent available also provides high surface area which is favorable for adsorption process (Mahajan and Sud, 2013) [11].

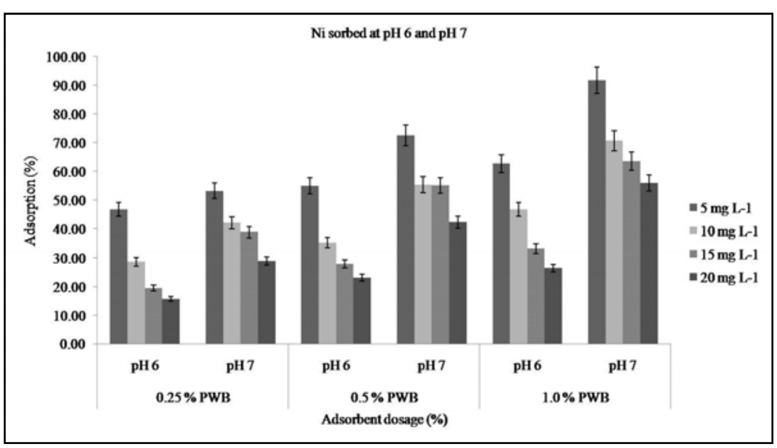

Fig 4: Effect of adsorbent dose on removal percent of metal ions at pH 6 and pH 7 (%)

4. Conclusion
The maximum removal efficiency of nickel by *Prosopis* wood biochar is 91.72 % at 5 mg L^{-1} at pH 7.0. The rate of sorption of nickel 3.05 mg g^{-1} increased significantly with increased concentration of nickel in aqueous solution until 20 mg L^{-1}. The adsorption of nickel was increased with increase the concentration of metal solution and biochar dose. The *Prosopis* wood biochar can be used as low cost adsorbent in for nickel removal effectively.

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