Role of Purged Air in the Synthesis of the Mesoporous NiO/C Composite and Its Application in Wastewater Treatment

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Abstract In this study, two methods were used to synthesize the NiO/C composite from agricultural waste. The mesoporous composite was successfully synthesized via a novel precipitation method in the presence of dissolved gases. The morphology of the composites was differentiated by using characterization techniques such as X-ray diffraction, the point of zero charge ($\text{pH}_{\text{pzc}}$), field-emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy, energy-dispersive X-ray analysis (EDAX), and vibrating sample magnetometry (VSM). Then, the mechanism of synthesis was elucidated using the above experimental characterization data. Results of FESEM and EDAX analyses of Ni(OH)$_2$–carbon composite clearly showed the role of dissolved gases in the synthesis. Both the composites were subjected as the adsorbent to remove the toxic Pb(II) ions from the wastewater. Batch adsorption experiments were carried out to compare the Pb(II) ion removal capability of both the composite materials. The parameters such as the effect of pH, the dosage of the adsorbents, and initial concentration were studied. At the optimized conditions, isotherm studies for each of the adsorbent were also carried out. The isotherm results revealed that the maximum removal capacity $q_e$ (mg/g) was 30.78 for PJNC and 43.48 for PJGNC. The VSM analysis confirmed that both the adsorbents were soft magnetic materials. Hence, they could be competently separated from salted/treated water using a magnetic field.

Keywords Role of dissolved gases · NiO/carbon · Carbon composite · Pb(II) ion removal · Heavy metal ions · Adsorption

1 Introduction

The earth is surrounded by three-fourths of water. Despite this statistical fact, there is more demand for potable water due to urbanization and industrialization (Luzardo et al., 2017). The heavy metal ions such as Pb(II), Cu(II), Hg(II), Cd(II), and Cr(IV) in aquatic streams credibly threaten the environment directly (Saranya et al., 2020; Sdiri & Higashi, 2013; Zhang et al., 2018). Research interests on the potential removal/subtraction of toxic metal ions from the
wastewater are focused on various methods such as chemical precipitation, coagulation, electrofloation, ion exchange, adsorption, photodegradation, and membrane filtration (Hua et al., 2012; Patra et al., 2020; Shaheen et al., 2018; Wan Ngah & Hanafiah, 2008). Among these, adsorption technique is more efficient, cost-effective, and eco-friendly. The economic adsorption process could be appropriately determined by the proper selection of the cheapest and the most effective adsorbent. Activated carbon is the most commonly used absorbent today as it is the most economical and efficient. This investigation focuses on the utility of the biowaste material, which is expected to perform better than the commercial activated carbon (Sud et al., 2008; Wan Ngah & Hanafiah, 2008). In this study, carbon was derived from *Prosopis juliflora* (PJ) wood, which is invasive to the environment. *P. juliflora* is usually found in abundance in the arid and semiarid continents (Chandrasekaran et al., 2020; El-Keblawy & Abdelfatah, 2014; Shackleton et al., 2015; Zachariades et al., 2011).

In the recent times, researchers have paid attention to convert nanometal oxides such as ferric oxides, aluminium oxides, manganese oxides, magnesium oxides, cerium oxides, and titanium oxides as an efficient adsorbent, as they have a large surface area and high activities with magnetic regeneration property (Bharath et al., 2021; Hua et al., 2012; Zachariades et al., 2011). Metal oxides are more effective, but they are not cost-effective. Latest studies reveal that metal oxide/carbon composites play a key role in high-power devices, electrochemical capacitors, catalysts, and adsorbents (Fu et al., 2019; Modwi et al., 2017; Wu & Hsieh, 2008) because the metal oxide/carbon composites are efficient and cost-effective. Hence, many efforts have been directed to synthesize an efficient carbon composite. The features of the composite have been determined by the method of synthesis (Hale, 1976; Zachariades et al., 2011). In this study, NiO/carbon composite was synthesized by precipitation method using sodium hydroxide and nickel nitrate. The PJ wood parts were collected from places in and around Coimbatore (11.0168° N, 76.9558° E), Tamil Nadu, India. The PJ wood was wrecked and broken into comparable similar sizes (2–3 cm) and later washed gently by using double-distilled water. The PJ wood pieces were taken in a muffle furnace and exposed to pyrolysis with a slow heating rate (5 K/min) (Estela et al., 2018). It was kept in the furnace up to 673 K to get a high yield of carbon (Selvaraju et al., 2018). The prepared carbon (named as *P. juliflora* carbon; PJC) from *P. juliflora* was washed thoroughly using double-distilled water to remove the ash and some dissolved matter. Then, it was dried and cooled to atmospheric temperature. The PJC obtained from the above process was crushed and converted into a composite with NiO by two precipitation methods.

### 2 Material and Methods

#### 2.1 Materials

The analytical-grade (AR) reagents such as nickel nitrate (Ni(NO3)2), lead nitrate [Pb(NO3)2], and sodium hydroxide (NaOH) were purchased from Merck Chemicals, Mumbai, India. Carbon was derived from a biological invasive matter (*Prosopis juliflora* wood).

#### 2.2 Synthesis of *P. juliflora* Carbon

The *P. juliflora* (PJ) wood parts were collected from places in and around Coimbatore (11.0168° N, 76.9558° E), Tamil Nadu, India. The PJ wood was wrecked and broken into comparable similar sizes (2–3 cm) and later washed gently by using double-distilled water. The PJ wood pieces were taken in a muffle furnace and exposed to pyrolysis with a slow heating rate (5 K/min) (Estela et al., 2018). It was kept in the furnace up to 673 K to get a high yield of carbon (Selvaraju et al., 2018). The prepared carbon (named as *P. juliflora* carbon; PJC) from *P. juliflora* was washed thoroughly using double-distilled water to remove the ash and some dissolved matter. Then, it was dried and cooled to atmospheric temperature. The PJC obtained from the above process was crushed and converted into a composite with NiO by two precipitation methods.

#### 2.3 Synthesis of NiO/C Composites

First, 4.6 g PJC was taken into a clean beaker along with 400 mL distilled water. After half an hour, that heterogeneous solution was constantly air purged at a flow rate of 2 × 10−2 m3/h. Then, 50 mL of 1 M NiNO3 was added to the above and the purging process continued about 30 min for even dispersion. To
this mixture, 50 mL of 2.0 M NaOH gradually was added at regular intervals, which resulted in a green Ni(OH)$_2$ precipitate on the surface of the carbon. That mixture was kept for a whole day for the settlement of the Ni(OH)$_2$/PJC composite (PJGNH) at the bottom. The filtrate was decanted and the residue was washed with double-distilled water to achieve the neutral pH. Then, the final product was kept in the muffle furnace and heated for half an hour up to 250 °C to get a black PJ shell consequential nanocomposite NiO/PJC (PJGNC) (Xing et al., 2004; Yuan et al., 2005). The synthesis procedure is shown in Fig. 1 and Eqs. (1) and (2). In addition, the NiO/PJC composite was prepared by the above-cited method without purging of the air. The obtained Ni(OH)$_2$/carbon was termed as PJNH and it was calcinated to convert into the NiO/carbon composite, which was termed as PJNC. This process is also shown in Fig. 1 and Eqs. (3) and (4).

**Synthesis I**

\[
PJC + O_2 + Ni(NO_3)_2 + 2 NaOH(aq) \rightarrow PJC - O_2 - Ni(OH)_2 \downarrow + 2NaNO_3(aq) \tag{1}
\]

\[
PJC - O_2 - Ni(OH)_2 \triangleleft NiO - PJNC + O_2 + H_2O \tag{2}
\]

**Synthesis II**

\[
PJC + Ni(NO_3)_2 + 2 NaOH(aq) \rightarrow PJC - Ni(OH)_2 \downarrow + 2NaNO_3(aq) \tag{3}
\]

\[
PJC - Ni(OH)_2 \triangleleft NiO - PJNC + H_2O \tag{4}
\]

2.4 Characterization

The morphology of PJGNC and PJNC was uniquely distinguished by using FESEM (SUPRA 55 VP-4132; Carl Zeiss), XRD analysis (SmartLab; Rigaku). The composition of the elements present in PJGNC and PJNC was differentiated and quantified by EDAX analysis. The variance in magnetic property of PJGNC and PJNC was studied using a 7410 series VSM (Lakeshore). The adsorption/desorption nitrogen isotherm in the P/P$_0$ range at 77.3 K (ASAP 2020 V4.02 H) was used to accurately differentiate the specific surface area and pore size of the PJGNC and PJNC.

2.5 Batch Mode Adsorption Studies

Using acceptable standards and methods, a comparison of the removal capacity of the prepared adsorbents was

![Fig. 1 Diagrammatic procedure for the synthesis of PJGNC and PJNC (4.6 g of PJC with 400 mL of distilled water, air flow rate: \(2 \times 10^{-2}\) m$^3$/h, 1 M NiNO$_3$ and 2.0 M NaOH, calcination temperature: 250 °C)]
conducted by batch adsorption studies. In this study, 100 mL optimal concentrations of lead (II) ions solutions were taken with a specified amount of adsorbents in the reagent bottles. Subsequently, using a mechanical shaker, they were agitated at 180 rpm at 303 K to elucidate the optimal dosage of the adsorbents in the adsorption process and the optimum pH from initial concentrations of Pb(II) ions. The role of the pH in the adsorption process was investigated by equilibrating 100 mL of 10 mg/L Pb(II) ion solution with 1.0 g/L dried adsorbents at various pH values between 1.0 and 8.0. The adsorbents ranging from 0.5 to 1.0 g/L were studied with a specified Pb(II) ion solution to find their optimal dose. The probability of the adsorption process was studied. The finest pH and the composite doses were accurately determined as 6.0 and 1.0 g/L for both PJNC and PJGNC. All the batch experiments were carried out with the abovementioned parameters with an equilibration time of about 180 min. After each batch experiment, the supernatant solution was decanted and analyzed by using an atomic absorption spectrophotometer (AAS-WFX-130; Systronics). Therefore, the amount of Pb(II) ions removal ($q_t$) (mg/g) could be calculated by following Eq. (5) and the adsorption efficiency ($R_t$) could be calculated using Eq. (6) (Shahnaz et al., 2020):

$$\text{Amount of Pb(II) ions removal } (q_t) \text{(mg/g)} = \frac{(C_i - C_e)V}{W}$$

where $C_i$ is the metal ions concentration measured before adsorption, $C_e$ is the metal ions concentration measured after adsorption, $W$ is the weight of the dried adsorbent, and $V$ is the aqueous solution volume in liters.

### 2.6 Isotherms Analysis

The unique design of the adsorption system could be correlated by the adsorption isotherm (Hameed et al., 2008; Saravanakumar et al., 2019). It would be necessary to explain the dispersion of adsorbate on the adsorbent in the liquid phase (Yao et al., 2016). The batch adsorption results in the present study were analyzed using two major isotherm models namely Freundlich isotherm and Langmuir isotherm. The first one indicated the equilibrium distribution of Pb(II) ions between the solid and liquid phases. This isomer was valid effectively for only monolayer adsorption onto a surface with a finite quantity number of active sites. This isotherm model assumed unchanging/uniform energies of adsorption onto the surface and no drifting/transmigration of adsorbate on the surface of the adsorbent (Bouabidi et al., 2018). Langmuir isotherm could be represented by following Eq. (7):

$$Q_e = \frac{Q_0b_LC_e}{1 + b_LC_e}$$

Linear form of Langmuir Eq. (8) could be represented as:

$$\frac{C_e}{Q_e} = \frac{1}{Q_0b_L} + \frac{C_e}{Q_0}$$

where $C_e$ is the equilibrium concentration of Pb(II) ions (mg L$^{-1}$), $Q_e$ is the amount of Pb(II) ions adsorbed per gram of the adsorbent at equilibrium (mg/g), $Q_0$ is the maximum monolayer coverage capacity (mg/g), and $b_L$ is the Langmuir isotherm constant (L/mg).

The Freundlich isotherm was used to describe the adsorption of metal ions on the heterogeneous surface. This isotherm does not necessitate limit of the adsorption when coverage is sufficient to fill a monolayer. It could be represented by following Eqs. (9) and (10) (Khozhaenko et al., 2016):

$$Q_e = k_F C_e^{1/n}$$

$$\log Q_e = \log k_F + \frac{1}{n} \log C_e$$

where $n$ is the adsorption intensity, $k_F$ is the Freundlich isotherm constant (mg/g), $Q_e$ is the amount of Pb (II) ions adsorbed per unit gram of the adsorbent at equilibrium (mg/g), and $C_e$ is the equilibrium concentration of adsorbate (mg/L).

### 3 Results and Discussion

#### 3.1 Structural Variation in PJNC and PJGNC

The structural variance of the PJNH and PJGNH composites could be understood by the powder XRD...
pattern, which is shown in Fig. 2. The $2\theta$ values of both PJNH and PJGNH are related to the respective Miller indices (001), (100), (101), (102), (110), (111), and (200) (Huang et al., 2007). The broadened peak of PJGNH and PJNH indicates poor crystallinity. The intensity of PJGNH is a little higher than PJNH. It shows the orientation effect of purged gases on the precipitation of nickel hydroxide. It supports high aggregation of Ni(OH)$_2$ with an outer layer of carbon particles. The reduced intensity peaks on PJNH indicate the domination of carbon on precipitation (Poinern et al., 2009). The crystalline structure of PJNC and PJGNC could be explained by the powder XRD analysis. Figure 3 shows the powder XRD analysis of PJNC and PJGNC. The $2\theta$ values of both PJNC and PJGNC are matching to the corresponding (111), (200), (220), (311), and (222) Miller indices (Mahmoud et al., 2015; Suresh et al., 2016; Wu & Hsieh, 2008; Xiang et al., 2002). The observed values denote the presence of nano-NiO crystallites in the composite. The calcination of PJC-NiOH at 250 $^\circ$C influences the orientation effect and changes the phase of amorphous carbon (PJC) to a crystalline composite. The peak intensity of PJGNC is higher than PJNC. It also indicates the extent of crystallinity of PJGNC (Inoue & Hirasawa, 2013). The sharp peaks of PJGNC show mesopore size enlargement. It clearly supports the formation of Ni(OH)$_2$ influenced by purged air. The continued purging of air tends to disperse properly of the carbon particles in the liquid phase on precipitation of Ni(OH)$_2$. It also leads to the quantized effect (Jayaram & Prasad, 2009).

3.1.1 FTIR Spectral Analysis

The reports of the FTIR analysis of PJNC and PJGNC are shown in Fig. 4. The PJNC and PJGNC composites were derived from *P. juliflora*. Therefore, they showed peaks of the functional group present in PJC. In Table 1, the observed peaks and presented functional groups are represented along with the assignments (Khalil et al., 2010; Pallarés et al., 2018; Saravanakumar et al., 2013; Shen & Gu, 2009). The wood-derived adsorbents comprised a combination of cellulose, hemicellulose, and lignin content. Hence, the composites PJNC and PJGNC showed peaks of the primary functional group of O–H at 3781 and 3785 cm$^{-1}$ (Tang et al., 2019). The peak at 1055 cm$^{-1}$ indicate the ester (–C–O–C–) assembly. Carboxyl stretching for acetyl C–O groups in aldehyde and hemicellulose groups of lignin was characterized by the existence of the peaks at 1691 cm$^{-1}$. The observed peaks at 1741, 1678, and 1067 cm$^{-1}$ confirm the occurrence of C–O, C=O, and C–O–C,
respectively (Khalil et al., 2010). The prominent peak around 590 cm\(^{-1}\) is attributed to the presence of NiO group in the composites (Suresh et al., 2016; Tang et al., 2019).

### 3.1.2 Zeta potential Studies

Generally, the pH of solution affects the surface charge on the adsorbent (Priya et al., 2018; Karthik et al., 2011). The point of zero charges (pH\(_{\text{pc}}\)) of the
adsorbent is one of the important factors to predict the range of pH, which shows maximum adsorption (Kosmulski 2009; Tang et al., 2019). The pH<sub>pzc</sub> can be calculated by the plot of pH vs zeta potential. According to Fig. 5, the pH<sub>pzc</sub> of PJNC and PJGNC calculated are 6.0 and 5.7, respectively. It is shown that PJNC has a neutral surface charge at pH (6.0) and a positive surface charge (zeta potential +23 to 3 mV) at pH (<6.0). Then, it shows a negative surface charge (zeta potential −5 to −29 mV) at pH>6.0. The zeta potential style for PJGNC slightly fluctuates and differs compared to PJNC.

Similarly, PJGNC had positive surface charge (zeta potential +19 to +4 mV) at pH<5.7, negative surface charge (zeta potential −2 to −34 mV) at pH>5.7, and neutral surface charge at pH 5.7. The pH<sub>pzc</sub> values of both PJNC and PJGNC were lower than those of pure NiO nanoparticles (pH<sub>pzc</sub> = 10.8), which confirm that the surface of NiO was impacted by PJC (Acharaya et al., 2009). The high pH<sub>pzc</sub> value of PJNC confirmed the decomposition of functional groups in the carbon of the composite and led to the decrease of the negative sites on the adsorbents. Meanwhile, the dissolved gases present in the PJGNH were influenced to avoid the decomposition of the functional group during calcination (Feygenson et al., 2010). It could be confirmed by the low pH<sub>pzc</sub> of PJGNC. The negative (−ve) surface of PJGNC influenced the positively charged Pb(II) ions and generated the interactions among Pb(II) ions and showed higher removal efficiency than PJNC.

### 3.1.3 BET Analysis and Particle Size of Adsorbents

The composite synthesis method was used to study the surface properties of the material. The Brunauer–Emmett–Teller (BET) model precisely revealed how metal oxide had combined with PJ carbon. The mean pore diameter (d), total pore volume (P/<sub>P</sub>o), and specific surface area (A<sub>BET</sub>) were calculated and the values are given in Table 2. The results show that the surface area and pore volume of PJNC are lower than those of PJGNC. The dioxygen in PJGNH at calcination increased the mean pore diameter of PJGNC. The average particle sizes of PJNC and PJGNC were calculated using a particle size analyzer (SZ100; Horiba, Japan) and reported as 71 ± 5 and 89 ± 5 nm, respectively. The particle sizes of both composites were higher than those of PJC (37 nm), which confirmed the aggregation of NiO with PJC.

### 3.1.4 SEM-EDAX Analysis

The scanning micrographs (FE-SEM) precisely differentiated the morphology of PJNH and PJGNH. Figure 6a and b show the aggregation of Ni(OH)2 with PJC. It reveals a high accumulation of Ni(OH)2 taking place in PJGNH due to the accessibility of more available active sites, which were influenced by the dissolved oxygen. The micrograph of Fig. 6c visibly confirms the gases between Ni(OH)2 and PJC. It is intentionally caused to increase the surface

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**Table 1** FTIR analysis report for PJNC and PJGNC

| Position of the peak (cm<sup>−1</sup>) | Assignments                          | Ref                        |
|--------------------------------------|--------------------------------------|----------------------------|
| 3781                                 | O–H stretching                       | Mahmoud et al., 2015       |
| 3468                                 | O–H stretching and N–H stretching   | (Modwi et al., 2017; Mousa et al., 2016) |
| 3051                                 | C–H stretching                       | (Pallarés et al., 2018; Patra et al., 2020) |
| 2292                                 | N–H bending                          | (Patra et al., 2020)       |
| 1691                                 | C=C, C = O stretching                | (Pallarés et al., 2018)    |
| 1463                                 | Aromatic C = C stretching            |                           |
| 1355                                 | C–N peptide bond                     |                           |
| 1194                                 | OH bending                           | (Poinern et al., 2009)     |
| 1068                                 | C–O–C                               | (Mousa et al., 2016; Patra et al., 2020) |
| 848                                  | Aromatic = C–H out-of-plane          | (Modwi et al., 2017)       |
| 591                                  | NiO stretching                       | (Kloss et al. 2012)        |
area and pore diameter of the PJGNC composite throughout the calcination process (Mahmoud et al., 2015). The micrographs in Fig. 6d and f show the structures of PJNC and PJGNC, respectively. Figure 6f shows the mesoporous structure of PJGNC, which is created by purged gases. Figure 6e and g show PJNC and PJGNC after adsorption of Pb(II) ions. It reveals that the high amount of Pb (II) ions adsorbed on the surface of PJGNC is due to the influence of the high surface area, pore diameter, and more active sites on the adsorbent (Tang et al., 2019).

The SEM-EDAX elemental dot maps of PJNH and PJGNH are shown in Fig. 7a. The light green, violet, and brown dots in the figure indicate the concentrations of O, C, and Ni, respectively. The presence of a higher luminous intensity of dots indicates a significant concentration of the element. In this mapping, the light green dots indicate that the quantity of oxygen distribution is highly abundant in PJGNH compared to PJNH. The EDAX analysis in Fig. 7b and c also confirmed the presence of a high quantity of dioxygen in PJGNH.

### 3.1.5 VSM Analysis

Generally, the potential removal of powder adsorbent from the effluent after treatment is very difficult. It could be made easy by using a magnetic adsorbent. The magnetic properties of nanocomposites were characterized using a VSM graph (Ahilandeswari et al., 2020; Gupta et al., 2011). From Fig. 8, the hysteresis loop of both the adsorbents reveals an antiferromagnetic character (Tang et al., 2018). This type of small hysteresis loops are meant to be the soft type magnets and complete magnetization enhances their low squareness shape. The magnetization values of PJGNC (87 × 10⁻³ emu/g) and PJNC (14 × 10⁻² emu/g) were found to be decreased compared to pure NiO (65 emu/g) due to the presence of nonmagnetic carbon. It confirmed

![Fig. 5 pH vs zeta potential (pH_{zpc} of PJNC is 6.0 and pH_{zpc} of PJGNC 5.7)]

![Table 2 BET Analysis of PJNC and PJGNC](data:image/png;base64,iVBORw0KGgoAAAANSUhEUgAAAgAAAAAgBAMAAAB...)

### Table 2 BET Analysis of PJNC and PJGNC

| Adsorbent | PJNC  | PJGNC  |
|-----------|-------|--------|
| Specific surface area (ABET) | 35 m²/g | 58 m²/g |
| Total pore volume (P/P₀) | 0.0182 cm³/g | 0.029 cm³/g |
| Mean pore diameter (d) | 1.96 nm | 2.05 nm |
| Particle size | 85 nm | 94 nm |
the successful composition of nonmagnetic carbon along with NiO. The value of coercivity in the composite PJGNHC was 125.65 and for PJNC, it was 113.95 G. These low coercivity values of both the adsorbents confirmed soft magnetic nature (Cai et al., 2015; Feygenson et al., 2010; Fu et al., 2019). The very low value of retentivity also signposted/indicated easier demagnetization of the prepared
composite (Ghaemi et al. 2017; Indhu & Muthukumarana, 2018). Hence, both could act as good adsorbents in the effluent remediation field.

3.1.6 Reverberation of Synthesis Process

Generally, the composite properties are influenced by the synthesis method. In this study, NiO/C composite was synthesized in two ways. Initially, carbon and NiNO₃ solution were purged with air. The dioxygen present in the air was dissolved in this solution and combined on the surfaces and micropores of the carbon in the liquid phase. It was influenced to combine the Ni²⁺ ions with PJ carbon. After adding NaOH solution, the Ni²⁺ precipitated along with dioxygen entrapped between Ni(OH)₂ and carbon.

Fig. 7  a EDAX elemental dot maps of PJNH and PJGNH. b EDAX analysis of PJNH and PJGNH
The entrapped oxygen is clearly shown in the FESEM micrographs (Fig. 6c). The EDAX analysis also supported the presence of high dioxygen content in PJGNH compared to PJNH by showing high luminous intensity and weight percentage of O₂. At calcination, the entrapped gases would leave in the form of CO, CO₂, and O₂. The released gases enlarged the micropores of the composite to mesopores. It was confirmed by the BET analysis. At the same time, it avoided the decomposition of the active functional group present in the PJ carbon. The functional group present in PJGNC enhanced the negative surface charge on the composite, which was also confirmed by the zeta potential study.

3.2 Performance of Pb (II) Removal

3.2.1 Effect of pH

The adsorption efficiency of an adsorbent usually depends on the pH of the solution (Gupta et al., 2011). The impact of pH on the adsorption efficiency was examined in a range of pH values, from 2 to 9. Figure 9 clearly shows that the removal efficiency of both adsorbents has decreased after pH 7 due to precipitation of Pb(II) ions (Tang et al., 2018). Below pH 2, PJNC and PJGNC did not perceive the significant amount of Pb(II) ions. It specifies the Pb(II) ion adsorption on adsorbent active sites entered by the hydrogen (H⁺) ions (Gerçel & Gerçel, 2007; Ghaemi et al., 2017). On increasing the pH, the adsorption efficiency of both the adsorbents increased due to decrease in the hydrogen ion concentration. On increasing the pH from 2 to 6, the removal percentage of PJNC and PJGNC adsorbents increased from 34 to 83% and from 36 to 93%, respectively. The adsorption capacity of PJNC and PJGNC attained a maximum at pH 6 due to the availability of the negative surface charge on the adsorbents. However, the efficiency of PJGNC was found to be significantly higher than PJNC due to the presence of more dynamically active sites on PJGNC.

3.2.2 Effect of Adsorbent Dosage

The impact of adsorbent dosage on the removal of Pb (II) ions is shown in Fig. 10. It denotes that the removal efficiency of both the adsorbents had increased abruptly with the increase in the adsorbent dosage. It was found that on increasing the adsorbent dose, the number of availability of active
sites also increased. Therefore, it favorably helped to increase the adsorption efficiency (Acharya et al., 2009). While increasing the weight of adsorbents from 0.5 to 10.0 g/L, the removal efficiency of PJNC increased from 76.69 to 95.68%. Similarly, the removal efficiency of PJNC improved from 73.59 to 93.58%, 63.81 to 83.48%, and 54.86 to 79.06% for 20, 30, and 40 mg/L Pb(II) ion concentration, respectively, at constant temperature (303 K) and at pH 6. Comparatively, adsorption capacity of Pb(II) on PJGNC increased from 88.12 to 98.54%, 86.34 to 97.42%, 84.43 to 95.22%, and 81.22 to 95.12% for 10, 20, 30, and 40 mg/L Pb(II) concentration ions, respectively, with the increase in the adsorbent doses from 0.5 to 10.0 g/L at constant temperature (303 K) and at pH 6.0. Figure 10 shows that PJGNC has a higher adsorption efficiency due to the influence of the high surface area, pore diameter, and pore volume of PJGNC (Jaiswal et al., 2015).

3.2.3 Effect of Initial Concentration

The removal efficiency of the adsorbent varied depending on the concentrations of the adsorbate (Jaiswal et al., 2015). The effect of concentration on modification was assessed in the concentration ranges from 4 to 40 mg/L. The outcomes in Fig. 11a and b demonstrate that change in the concentration of adsorbate exerted a significant influence on the adsorption. The Pb(II) ion removal efficiencies (mg/g) of PJNC and PJGNC were improved on increasing the metal ion concentration, at the optimized pH and 1.0 g/L dosage.

While using the initial concentration from 4 to 40 mg/L, the adsorption capacities (mg/g) of PJNC and PJGNC were increased from 3.65 to 25.18 mg/g and 3.86 to 33.58 mg/g, respectively. The increasing trend of both the adsorbents confirmed that the absorption of Pb(II) ions on adsorbent was simply physical adsorption. At the same time, the removal percentage of Pb(II) ions decreased with the increase in concentration (Fig. 11a). Thus, it showed the presence of the specific/precise limit of adsorption sites on the surface of adsorbent (Ahrouch et al., 2019;
Goel et al., 2005; Khandanlou et al., 2015). The presence of a higher number of significant active sites and large pore diameter had significantly influenced the removal capacity of PJGNC than PJNC.

3.2.4 Isotherm Pattern

The efficiency of adsorption is usually determined by affinity and surface properties of the adsorbents toward the adsorbate. The nature of the surface of the adsorbent in the adsorption process could be effectively determined by isotherm patterns (Goel et al., 2005; Mousa et al., 2016). In this study, Langmuir and Freundlich isotherms were applied to show the nature of adsorption with the schematic mechanism of Pb(II) ions on the PJNC and PJGNC adsorbents. The isotherm marks can be derived from Fig. 12a and b and Fig. 13a and b. These are also illustrated in Table 3. It clearly exhibits that the removal of Pb(II) ions followed the Freundlich model more reasonably (Shi et al., 2019; Su et al., 2009). The maximum removal efficiency values of PJNC and PJGNC at 303 K were 30.78 and 43.48 mg/g, respectively.

The correlation coefficient (R2) of both the isotherms existed between 0.97 and 0.99. However, the Freundlich isotherm was more fitted over the entire temperature (Yang et al., 2018). The hexagonal Pb(II) ions on adsorbents in the SEM micrograph (Fig. 6g) also supported the multilayer formation. Thus, the following were demonstrated from the Freundlich isotherm:

- The multilayer exposure coverage was outward between the adsorbate and adsorbent at a persistent constant temperature (Li et al., 2018)
- \( 1/n \) value was below one (\( 1/n < 1 \)), which showed reversible physisorption (Dada et al., 2012), and
- The superior adsorption capacity of PJGNC was confirmed by a higher \( K_f \) value (Shahnaz et al., 2021).

The nature of the reactions was specified by using the \( R_L \) value, which was obtained from Langmuir isotherm. It also specified the shape of the isotherm to be irreversible (\( R_L = 0 \)) or unfavorable (\( R_L > 1 \)) or favorable (\( 0 < R_L < 1 \)) and linear (\( R_L = 1 \)). In this study, \( R_L \) values were placed between \( 0 < R_L < 1 \), which also confirmed that the adsorption was a favorable process (Habtegebrel & Khan, 2018). Comparing the isotherms models, this collegial adsorption process involved both monolayer and multilayer adsorption with a fraction of active
Fig. 11  a Effect of Pb(II) ion concentration (temperature: 303 K, adsorbent dose: 1 g/L, contact time: 3 h, pH: 6 for both PJNC and PJGNC). b Effect of Pb(II) ion concentration (temperature: 303 K, adsorbent dose: 1 g/L, contact time: 3 h, pH: 6 for both PJNC and PJGNC).
sites (Yang et al., 2018). These results concluded that PJGNC was a more favorable adsorbent than PNC in the removal of Pb(II) ions from the aqueous solutions.

4 Conclusions

The surface enhancement of PJNC and PJGNC composite was evidently investigated in this study.
Pb(II) ion removal efficiency of adsorbents synthesized using two processes was investigated. The novel preparation of the composite gave a higher pore diameter, pore volume, and surface area. In addition, carbon derived from agricultural invasive...
Table 3  The PJNC and PJGNC isotherm results  
(temperature: 303 K, 313 K, and 323 K; adsorbent dose: 1 g/L contact time: 3 h, pH: 6) 

|                | PJNC  | PJGNC |
|----------------|-------|-------|
|                | 303 K | 313 K | 323 K | 303 K | 313 K | 323 K |
| Langmuir isotherm | $b_L$ (L/mg) | 0.285 | 0.289 | 0.306 | 0.491 | 0.554 | 0.382 |
|                 | $q_m$ (mg/g) | 30.769 | 27.712 | 24.631 | 43.478 | 46.083 | 40.984 |
|                 | $R^2$ | 0.9835 | 0.9906 | 0.9892 | 0.9777 | 0.9379 | 0.9914 |
|                 | $C_e$ (mg/L) | $R_L$ | $R_L$ | $R_L$ | $R_L$ | $R_L$ | $R_L$ |
| 10              | 0.2598 | 0.2570 | 0.2463 | 0.169 | 0.153 | 0.208 |
| 20              | 0.1493 | 0.1475 | 0.1405 | 0.092 | 0.083 | 0.116 |
| 30              | 0.1048 | 0.1034 | 0.0982 | 0.064 | 0.057 | 0.080 |
| 40              | 0.0807 | 0.0796 | 0.0755 | 0.048 | 0.043 | 0.061 |
| Freundlich isotherm | $1/n$ | 1.890 | 1.978 | 2.092 | 0.5913 | 0.586 | 0.5993 |
|                 | $K_F$ (mg/g) | 6.663 | 6.183 | 5.829 | 12.272 | 14.018 | 9.954 |
|                 | $R^2$ | 0.992 | 0.990 | 0.980 | 0.9929 | 0.9955 | 0.9885 |

PJ wood was converted into a cost-effective nanocomposite. The XRD peak intensity revealed the extent of crystallinity and porous nature of PJGNC. At calcination of PJGNH, surface decomposition of the functional group was avoided by the presence of purged dissolved oxygen. It was visibly observed in the EDAX analysis. The anionic active –NH, C≡C, –OH, C–O sites, and phenolic and aromatic groups of both adsorbents were confirmed by the FTIR analysis. The FESEM micrographs of PJGNH confirmed the existence of gas molecules in between Ni(OH)$_2$ and carbon. The mesoporous nano-PJGNC was evidently identified by FESEM micrographs. The FESEM micrographs also specified the addition of the excessive amount of Pb(II) ions onto the PJGNC compared to PJNC. The mechanism of synthesis was found to prove the surface enhancement of the PJGNC. The VSM analysis also confirmed the highly soft magnetic character of PJGNC. The Langmuir and Freundlich isotherms were studied to investigate the surface nature of the adsorbents. The isotherm results revealed that the maximum removal $q_e$ capacity was 30.78 mg/g for PJNC and 43.48 mg/g for PJGNC. The Langmuir and Freundlich isotherm equations were studied and it was found that they jointly supported the multilayer physical adsorption existing between Pb(II) ions and the adsorbent. PJGNC gave high removal capacity compared to PJNC due to the high surface area, pore diameter, and pore volume. This study revealed that PJGNC could be used to treat the wastewater for efficient removal of Pb(II) ions.

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