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## Preparation, Characterization and Application of New Low Cost Adsorbents for Adsorption of Nickel Metal

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**ABSTRACT:** The current study is spotlighted with the evaluation of three low cost adsorbents like sulphonated Prosopis juliflora carbon (SPJC), sulphonated Hibiscus rosa-sinensis carbon (SHRSC) and sulphonated Psidium guajava carbon (SPGC) in the removal of Ni (II) ions from aqueous solution. The current materials before and after adsorption were suspiciously analyzed by scanning electron microscope (SEM), Fourier transform infra red (FT-IR), thermo gravimetric analysis (TGA) and differential thermal analysis (DTA). The upshots of contact time, pH, temperature, adsorbent dose, initial concentration were investigated for the adsorption of Ni (II) from polluted aqueous solution. The records for the adsorption of Ni (II) were tested with Freundlich and Langmuir adsorption isotherm model and then the process followed the Langmuir isotherm model by the found high  $R^2$  value.

**KEYWORDS:** Sulphonated Prosopis Juliflora Carbon; Sulphonated Hibiscus rosa-sinensis Carbon; Sulphonated Psidium Guajava Carbon; Nickel (II); Adsorption.

### I. INTRODUCTION

In our nation, nearly more than 50,000 big, average and petite level electroplating industries generally distributed in urban regions [1]. Electroplating industries require low volume of water for its operation compared to other industries, but the producing effluent is extreme poisonous than other effluents. Then, these industries create poisonous perilous ravage containing heavy metals which are harmfully affect environment, principally plants, aquatic life, humans and animals [2]. While waste water comprising heavy metals runs above the surface of the soil as it loses fertility and the waste water holding nickel, begins mainly from metal industries, predominantly through plating operations. Happening of eczema in several workers employed in electroplating, cleaning paints and pigments may be assigned to nickel poisoning [3]. Heavy metal perniciousness was created the damage to central nervous function, blood composition, lungs, kidneys, liver and organs [4&5]. If heavy metals ion concentration outgoes permissible limits, it can create health ventures. Hence, wastewater should be treated before passes throughout water bodies or land. Keyed as industries like nuclear power operation, battery manufacturing, dye, electroplating, mining and pigment removing sewage have lofty level of Ni (II) ions [6]. Presently several different physical and chemical techniques have been utilized to removal of Ni (II) from effluents like reverse osmosis, chemical precipitation, membrane filtration, ion exchange, electrochemical reduction, evaporation, solvent extraction and adsorption [7]. Although in budding countries, these methods are still too lavish to be utilized. Nevertheless adsorption is a recovered technique among these methods for the waste water treatment [8], because competent and cheap. Some researchers have applied the low cost adsorbents as coconut shell, waste tea powder, coconut husk, sugar cane waste, saw dust, rice husk, coir pith etc [9]. In the current study, low cost adsorbents such as SPJC, SHRSC and SPGC applied for the removal of Ni (II) ions from aqueous solution. These three adsorbents were grinded and analyzed structurally as well as thermally with various spectroscopes and subsequently applied as adsorbents in optimal conditions for the elimination of Ni (II) from Polluted aqueous solution [10].

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## II. EXPERIMENTAL METHODS

The flora materials applied in the present study like SPJC, SHRSC and SPGC (Fig.1) are generously presented in Tamil Nadu, India. LR grade (purity: 98.3%) of concentrated Sulphuric acid (Sp.gr. = 1.82) was used for the preparation of activated carbon. The other chemicals and reagents were of chemically pure grade (AnalaR) purchased from SD Fine Chemicals, India.

Taken three adsorbents in the present research work were weighed alone as 300g. The weighed three adsorbents were carbonized and sulphonated with the addition conc.H<sub>2</sub>SO<sub>4</sub> gradually and kept at room temperature (30±1°C) for 24 h. Then these adsorbents were heated to 90°C in a water bath for 6h. After cooling, these materials were rinsed numerous times with water to eliminate free acid (tested with BaCl<sub>2</sub> solution) and dried at 70°C for 24h. These were consequently powdered, cleaned with distilled water and lastly with double distilled (DD) water to confiscate surplus free acid, dried, sieved (250– 300µm) by using Jayant sieves (India) and protected for characterization and these were called as SPJC, SHRSC and SPGC [11].

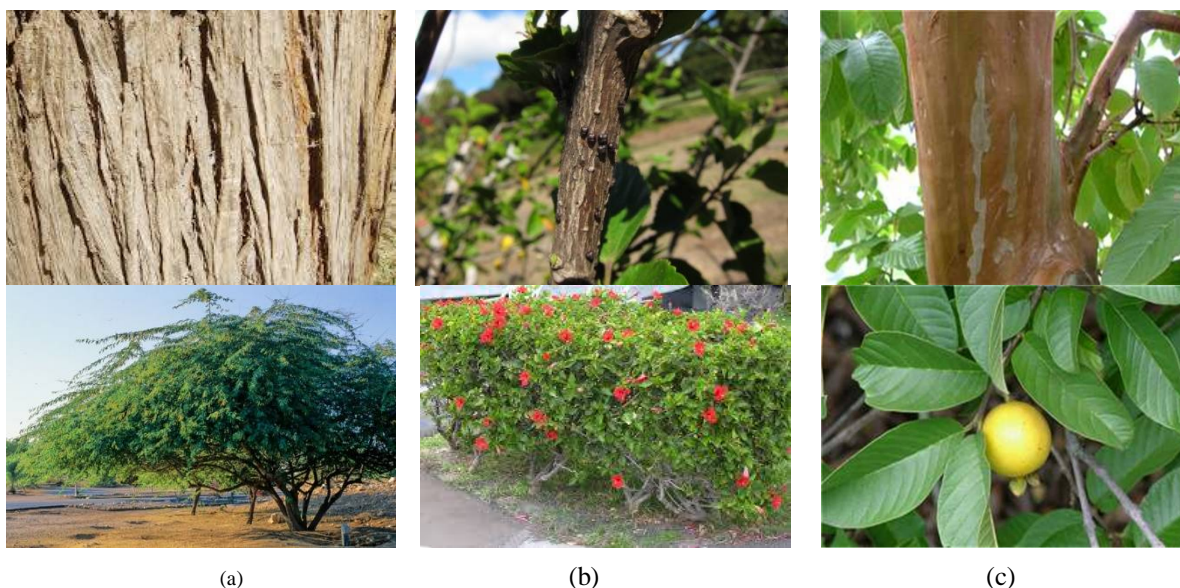


Fig.1 Raw Materials for the preparation of activated carbon. Bark of (a) Prosopis juliflora (b) Hibiscus Rosa Sinensis (c) Psidium Guajava

The equilibrium adsorption capacity ( $q_e$ ) of the adsorbents was estimated with the help of following equation:

$$q_e = (C_o - C_e) \times V / M \quad \text{----- (1)}$$

Where  $q_e$  is the equilibrium adsorption capacity ( $\text{mg g}^{-1}$ ),  $C_e$  is the concentration of metal ion ( $\text{mg L}^{-1}$ ) at equilibrium,  $V$  is the volume of solution (L) and  $M$  is the weight (g) of adsorbent.

The percentage removal of dye was calculated by using equation.

$$\text{Percentage removal} = (C_o - C_e) \times 100 / C_o \quad \text{----- (2)}$$

## CHARACTERIZATION OF THE SAMPLES

In the current research work, FT-IR (SHIMADZU MODEL FT-IR spectrometer) spectra was exploited to the examine the before and after Ni (II) adsorption on the three adsorbents by using the IR-grade KBr pellets in the ratio of 1:200 at the wave number ranging from 400 to 4000  $\text{cm}^{-1}$ . The sound structure of free and Ni (II) loaded adsorbents were

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studied with SEM (Vega3 Tescan SEM instrument). TGA and DTA analyzer (SII MODEL 6000 thermal analyzer) were employed to found out the thermal degradation of the free and Ni (II) loaded adsorbents.

## III RESULTS AND DISCUSSION

### SPECTRAL CHARACTERIZATION – FT – IR

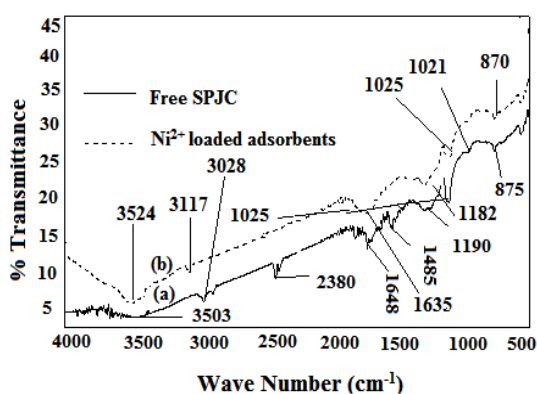


Fig.2 (a) & (b) IR spectra of the free and Ni<sup>2+</sup> loaded SPJC

The FT-IR spectra of the free SPJC and Ni (II) loaded SPJC were shown in Fig.2. These indicated a transfer in wave number of principal peaks related with the loaded metal and proved the metal binding process happening at the exterior of adsorbents [12]. This data provides evidence that the functional groups of SO<sub>3</sub><sup>-</sup> are involved in binding the Ni (II) ions onto the adsorbents.

### SEM ANALYSIS

SEM images of the free and Ni<sup>2+</sup> loaded adsorbents are demonstrated in Fig.3a & b correspondingly. As detected from figures, a detectable variation of the surface morphology in the Ni (II) loaded SPJC revealed that the adsorption of Ni (II) ions has occurred on the adsorbents.

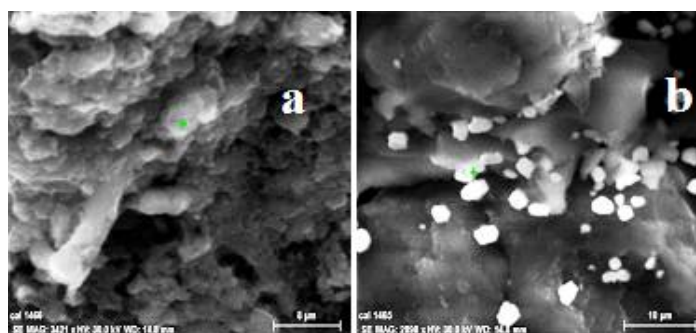


Fig.3 (a) & (b) SEM analysis of the free and Ni<sup>2+</sup> loaded SPJC

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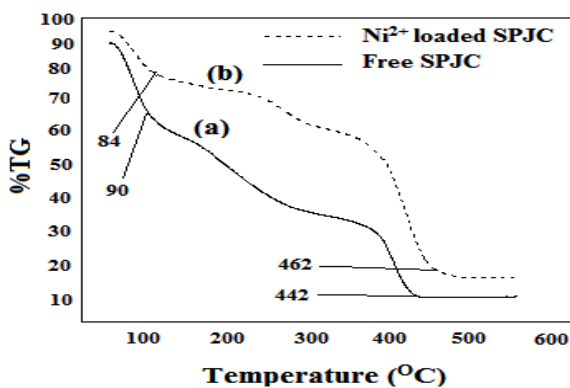
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## TGA AND DTA ANALYSIS

The TGA (Fig.4a & b) & DTA (Fig.5a & b) analysis of the free and Ni<sup>2+</sup> loaded SPJC are showed a transfer in temperature principal peaks associated with the loaded metal. This transfer in the temperature supports that the metal binding process happening at the surface of SPJC.



analysis of free and Ni<sup>2+</sup> loaded SPJC

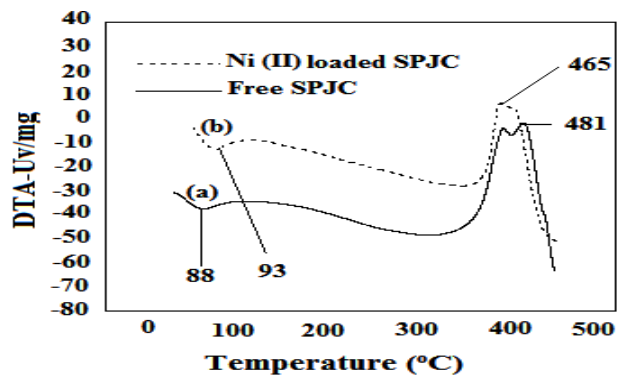


Fig.5 (a) & (b) DTA analysis of free and Ni<sup>2+</sup> loaded SPJC

Fig.4  
(a) &  
(b)  
TGA

## OPTIMIZATION OF ADSORBENT DOSAGE, PARTICLE SIZE AND p<sup>H</sup>

As found from the Fig.6, The premier adsorption capacity was examined for the adsorbent dosage of 30 mg/L. Fig. 6 is then indicated that the % of adsorption of Ni (II) increased with increasing adsorbent amount from 1 to 30 mg/L due to the enhance in adsorbents site available for the adsorption of Ni (II) ions. Afterward there is not considerable raise in the % of adsorption after 30 mg/L since moves constantly from 30 mg/L to 70 mg/L due to dissemination of adsorption sites in the adsorbent. The upshot of particle size on the adsorption of Ni (II) ions onto adsorbents was explored at particle size 200-300 micron. Fig.7 is obviously pointed out that the 250–300 micron was determined as the optimum as well as was employed in further experiments. As viewed from Fig.8, the maximum adsorption of Ni (II) onto adsorbents was reached at pH 4 due to the electrostatic attraction between adsorbents and adsorbate increases from p<sup>H</sup> 1 to 4. After that point, the adsorption process was decreased due to weakening of electrostatic force of attraction between them [13].

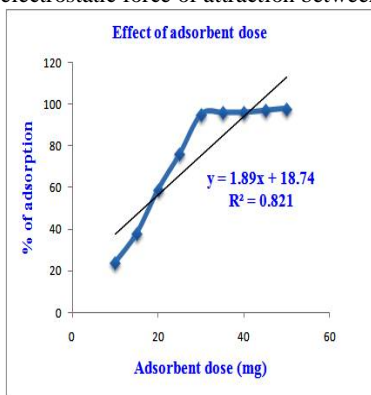


Fig.6. Effect of adsorbent dosage

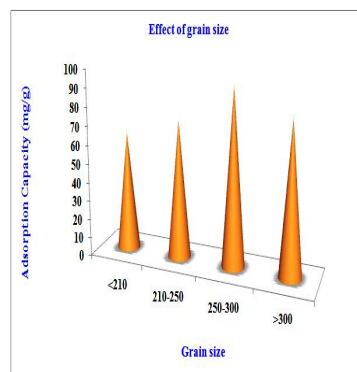


Fig.7. Effect of particle size

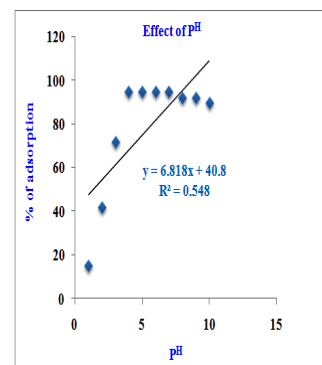


Fig.8. Effect of p<sup>H</sup>

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## EFFECT OF CONTACT TIME AND INITIAL NI (II) CONCENTRATION ON THE NI<sup>2+</sup> RETENTION

Fig.9 informs that the time dependant performance of the removal of Ni (II) from aqueous medium by the adsorbents. This shows evidently that the exclusion magnitude of Ni (II) increased as the contact time onwards. The equilibrium time was reached in 30 min. The crucial adsorption rate was very fast which conceivably due to the survival of more number of adsorbent sites existed for Ni<sup>2+</sup> elimination from aqueous solution. At the same time as the remaining presented surface areas decreases, the adsorption rate mired may be due to the progress of repulsive forces between the Ni (II) ions on the solid surface and in the liquid phase. Hence, no considerable development in the adsorption performance after 30 min. Fig. 10 showed that the adsorption of Ni (II) onto the adsorbents has been raised with raise in the initial Ni (II) concentration in the range 30 to 230 mg L<sup>-1</sup> by reason of the raise in driving force rooted by the progress of concentration gradient between the Ni (II) solution and surface of the adsorbents. Hence the built up adsorbents can be proficiently applied for the exclusion of high concentration Ni (II) from aqueous solution [14].

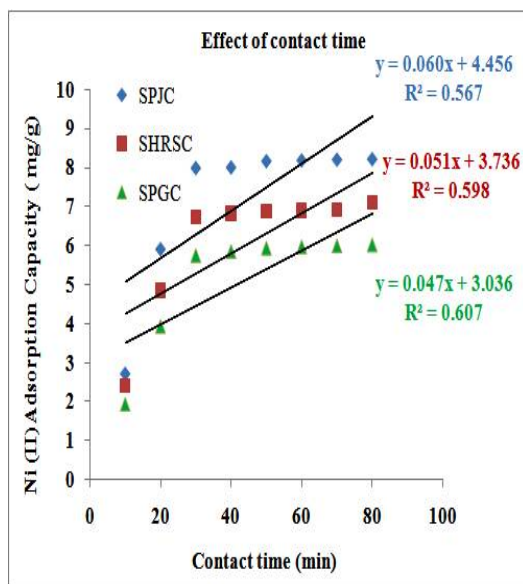


Fig.9. Effect of contact time

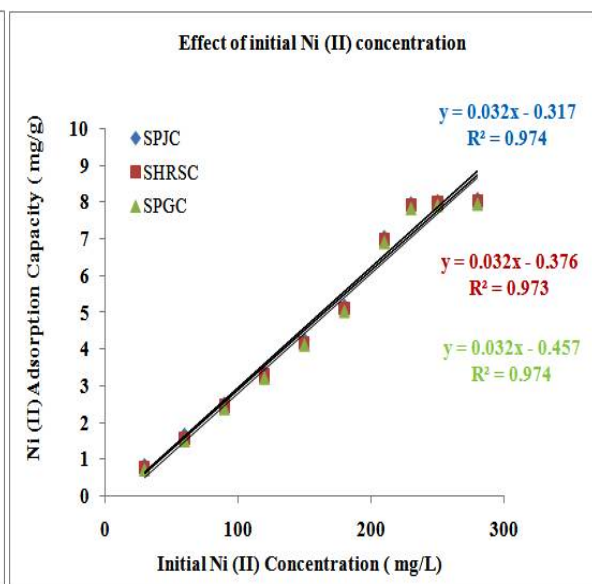


Fig.10. Effect of initial concentration

### ADSORPTION ISOTHERMS

The hold between adsorbents and Ni (II) can be calculated with the various adsorption isotherm models. The analyzed values from the adsorption of Ni (II) onto the adsorbents jibed to the linear equation of Langmuir and Freundlich isotherm models. The linear equation for Langmuir and Freundlich isotherm models are uttered as follows:

$$\text{Freundlich isotherm} \quad : \log q_e = \log K_F + (1/n) \log C_e \text{ ----- (3)}$$

$$\text{Langmuir isotherm} \quad : (C_e / q_e) = (1 / Q_o b) + (C_e / Q_o) \text{ ----- (4)}$$

Where  $K_F$  ( $\text{mg g}^{-1}$ ) is the Freundlich constant and 'n' the freundlich exponent. Where  $q_e$  ( $\text{mg g}^{-1}$ ) is the adsorbed amount of Ni (II) at equilibrium,  $C_e$  ( $\text{mg L}^{-1}$ ) is the equilibrium concentration of Ni (II),  $Q_o$  ( $\text{mg g}^{-1}$ ) and  $b$  ( $\text{L mg}^{-1}$ ) are Langmuir constants described to adsorption capacity and energy of adsorption. Additionally, the major features of the Langmuir isotherm can be explicated with separation factor  $R_L$ , which is analyzed with the equation 5.

$$R_L = 1 / (1 + bQ_o) \text{ ----- (5)}$$

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From the fig.11, 12 and 13 (a) & (b), the all values are calculated and tabularized in table 1 & 2. Langmuir isotherm models were fitted well while compared to Freundlich isotherm model for the Ni<sup>2+</sup> adsorption by three adsorbents through estimated R<sup>2</sup> value. The three adsorbents are fitting well for the removal of Ni (II) from aqueous solution by calculated n values has greater than 1. The R<sub>L</sub> value of three adsorbents range from 0 to 1 corroborated that the adsorption process is sympathetic for the adsorbents. Then in the present work, the found outcomes indicated clearly that the SPJC was advanced adsorbent likened to the former adsorbents. Earlier results reported in the various research works for the Ni<sup>2+</sup> adsorption are analogous to the reported adsorbent in the present work.

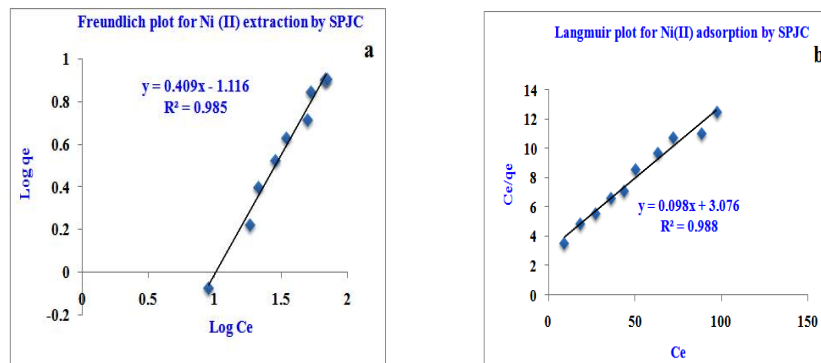


Fig.11 (a) & (b) Freundlich & Langmuir plots for Ni (II) removal by SPJC

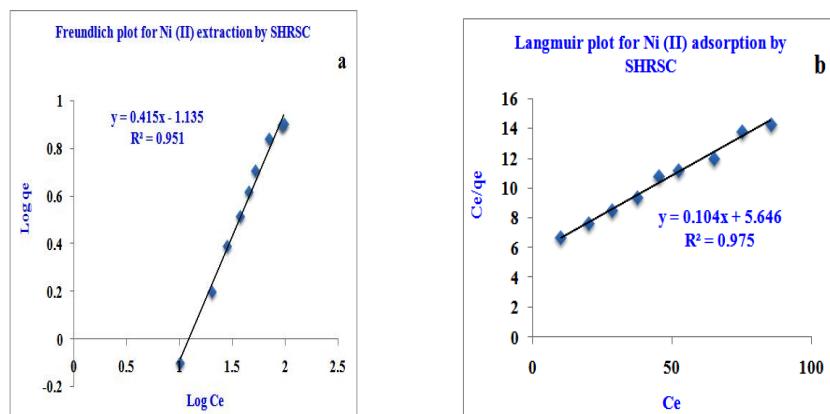


Fig.12 (a) & (b) Freundlich & Langmuir plots for Ni (II) removal by SHRSC

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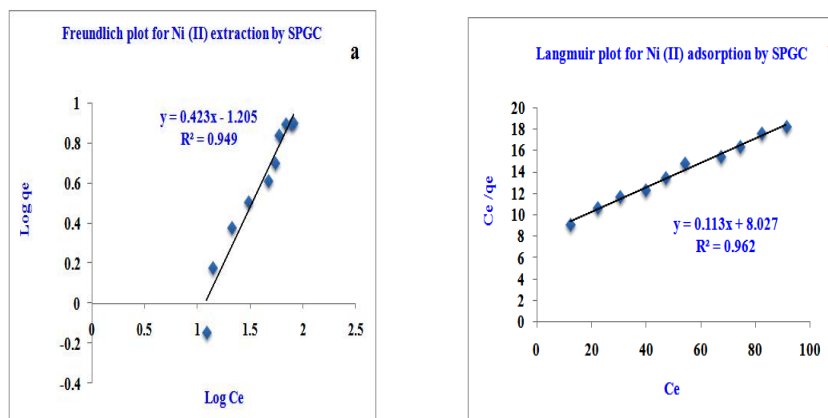


Fig.13 (a) & (b) Freundlich & Langmuir plots for Ni (II) removal by SPGC

**Table 1 Freundlich adsorption isotherm parameters**

Metal Ion	Freundlich								
	SPJC			SHRSC			SPGC		
	n	K <sub>F</sub>	R <sup>2</sup>	n	K <sub>F</sub>	R <sup>2</sup>	n	K <sub>F</sub>	R <sup>2</sup>
Ni (II)	2.4449	0.0765	0.985	2.4096	0.0732	0.951	2.3640	0.0623	0.949

**Table 2 Langmuir adsorption isotherm parameters**

Metal Ion	Langmuir											
	SPJC				SHRSC				SPGC			
	Q <sub>o</sub> (mg/g)	b (g/l)	R <sub>L</sub>	R <sup>2</sup>	Q <sub>o</sub> (mg/g)	b (g/l)	R <sub>L</sub>	R <sup>2</sup>	Q <sub>o</sub> (mg/g)	b (g/l)	R <sub>L</sub>	R <sup>2</sup>
Ni (II)	10.204	0.031	0.754	0.988	9.615	0.018	0.849	0.975	8.849	0.014	0.889	0.962

## IV. CONCLUSION

The adsorption performance of Nickel on these three adsorbents was examined in batch equilibrium adsorption. The adsorption was detected to be significantly reliant on pH, adsorbent dosage and contact time. The optimum pH for Ni ion was detected as 4. The tempo of Ni (II) adsorption was quick using three adsorbents with equilibrium contact time of 30 minutes. The adsorption between Ni (II) and three adsorbents are thermodynamically favourable and exothermic in nature. The exhausted adsorbents are efficiently renewed with 13BV of 10% (w/w) NaCl solution. The values calculated from the isotherm analysis indicated that the adsorption model of Ni (II) onto the three adsorbents

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followed by the Langmuir isotherm pattern. Kinetics information is corroborated with Pseudo second order reaction rate model.

The current study concludes that in all aspects the SPJC adsorbent could be applied as superior adsorbent than the other two adsorbents for the removal of Ni (II) from electroplating and other industrial effluent.

## V. ACKNOWLEDGEMENT

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