MJSAT

Malaysian Journal of Science and Advanced Technology

journal homepage: https://mjsat.com.my/

Impregnation of Single-Walled Carbon Nanotubes (Swcnts) with N-(3-Methoxybenzoyl)-N'-(Salicylidene) Thiosemicarbazone for Cobalt (Ii) Remediation from Aqueous Solution

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KEYWORDS

Single-walled carbon nanotubes
Impregnation
Hydrothermal retort

ARTICLE HISTORY

Received 22 November 2025
Available online 21 August 2026

ABSTRACT

Carbon nanotubes have capture attention in recent times due to their advance physicochemical properties which proven to be good on strength, flexibility, sensors, adsorption and conducting. The contamination of aquatic environments with heavy metals presents serious ecological risks, particularly due to the persistence and bio accumulative nature of these pollutants. Among them, cobalt (II) is growing concern, necessitating the development of efficient and sustainable remediation strategy. This study explores the synthesis and application of single-walled carbon nanotubes functionalized with thiosemicarbazone derivative as high-performance adsorbents for cobalt (II) removal from aqueous systems. A ligand was synthesized through a controlled multi-step process involving benzoyl thiocyanates, thiosemicarbazone and an aromatic aldehyde. This ligand was subsequently grafted onto SWCNTs surface using a hydrothermal-assisted retort method to enhance their surface reactivity, dispersion ability, and metal-binding efficiency. The compound was experimentally characterized by using FTIR, NMR, SEM-EDX, and ICP-OES. Batch adsorption experiment revealed that the functionalized SWCNTs has a strong affinity for cobalt ions with optimal removal efficiency observed at pH 6.0. It reached adsorption equilibrium within 50 minutes, and the data was best described by Langmuir isotherm model (R2 > 0.99) suggesting monolayer adsorption, followed by pseudo-second-order kinetics, indicating chemisorption mechanism. Specifically, SWCNTs functionalized with methoxy substituted ligand showed superior performance, likely due to increased electron-donating effects enhancing metal-ligand interaction

1. INTRODUCTION

Cobalt is a trace metal that can be found in water, typically due to natural processes like weathering rocks or from industrial activities which usually found in form of gas, liquid or solids. High concentrations can lead to adverse health effects, such as cardiovascular problems and neurological issues [1]. In recent years, presence of cobalt in wastewater has increased substantially due to its extensive use in emerging technologies. Hence, finding the most cost-effective and efficient methods of removing heavy metals from wastewater is crucial [2]. Thus, the development of efficient and sustainable methods for cobalt (II) removal from aqueous systems has gained considerable attention.

Among various water treatment techniques, adsorption is recognized as one of the most effective and economical approaches owing to its simplicity, high efficiency and adaptability [3]. Carbon-based nanomaterials particularly carbon nanotubes have attracted significant interest as adsorbents due to their high surface area, chemical stability and ease of surface functionalization [4]. Single-walled carbon nanotubes (SWCNTs), offer superior adsorption performance due to well-defined structure and accessible active site [5].

Functionalization of SWCNTs with selective chelating agents is an effective strategy to enhance their adsorption capacity and selectivity towards specific metal ions. Thiosemicarbazone derivative is well known for their strong

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https://doi.org/10.56532/mjsat.v6iS1.795

metal-binding ability through donor atoms such as nitrogen and sulphur, enabling stable coordination with transition metal ions [6][7]. In this study, a methoxy-substituted thiosemicarbazone ligand was designed to further enhance metal-ligand interaction. The electron-donating methoxy group increases electron density on the coordinating sites, thereby strengthening cobalt (II) complexation and improving adsorption efficiency [8]. Hence, to achieve effective impregnation of ligand onto SWCNTs, a hydrothermal-assited retort method was employed. Compared to conventional methods, this technique offers improved dispersion, enhance surface reactivity and stronger interfacial interaction between the ligand and SWCNTs [4]. These advantages overcome common challenges such as agglomeration and non-uniform functionalization which often limit adsorption performance

Therefore, this work focuses on the synthesis, characterization and adsorption performance of SWCNTs impregnated with a methoxy-substituted thiosemicarbazone ligand for cobalt (II) removal from aqueous solutions. The effects of pH, contact time and adsorption kinetics were systematically investigated to evaluate the potential of this material as an efficient adsorbent for industrial wastewater remediation.

2. METHODOLOGY

2.1 Synthesis of Benzoyl Thiocyanate using 3- Bromobenzoyl Chloride

The 3-Bromobenzoyl Chloride (1.2ml, 10mmol) was added dropwise to a stirring acetonitrile solution (40ml with ammonium thiocyanate (0.76g, 10mmol). White precipitates of ammonium chloride, NH4Cl were formed immediately and filtered. The filtrate was added dropwise into a stirring thiosemicarbazone (0.91g, 10mmol) with 40 ml acetonitrile. Next, the solution was refluxed for 4 hours and 90 °C with constant stirring.

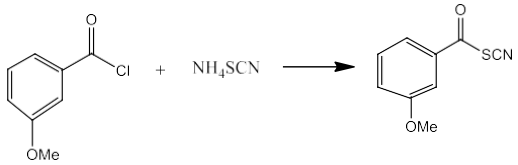

Fig. 1. The first step of synthesis process.

2.2 Synthesis of ligand

Salicylaldehyde (1ml, 10mmol) was added dropwise to a stirring acetonitrile solution (40ml) into mixed solution and continue refluxed for 2 hours with constant stirring. Then, the solution was poured into a beaker containing some ice cubes. The precipitate was filtered and washed by cold methanol and dried in desiccator for 24 hours.

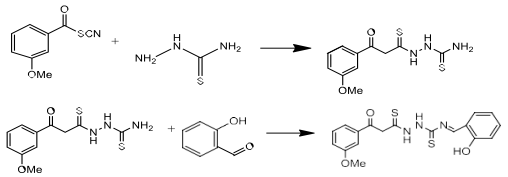

Fig. 2. Shows the synthesis of ligand N-(3- methoxybenzoyl)-N'(salicylidene)-thiosemicarbazone.

2.3 Impregnation of single-walled carbon nanotubes with ligand.

The amount of single-walled carbon nanotubes SWCNTs was weighed and added into 50ml of ethanol in sealed vial. The mixture was placed in hydrothermal-assisted retort condition for 1 hour under constant pressure, 24 PSI. In a quantity ratio of 1:1, 1g of powder ligand was added into 1ml of single-walled carbon nanotubes mixture and refluxed for another 4 hours at 90 °C. The sample was filtered and dried in a hot oven at 100 °C and kept in desiccator for characterization.

Fig. 3. The retort process of SWCNTs-ligand mixture.

2.4 Material Characterization

FTIR Spectra were recorded to identify functional groups within ligand and modified SWCNTs. ^{1}H NMR spectroscopy (CD3OD) deuterated methanol solvent was used to confirm ligand structure. Surface morphology element composition was analysed using SEM-EDX. Cobalt concentrations were determined using ICP-OES.

2.5 Adsorption study

The adsorption isotherm for Co^{2+} ion was carried out by preparing five different concentrations of 10, 20, 30, 50 and 80 ppm of Co^{2+} ions stock solution. The isotherm data were correlated with the Langmuir and Freundlich model. The quantities of adsorbed metal ions were calculated using equation 1 and 2 [3]:

$$qt = \frac{(co - ct)}{m}.V \quad (1)$$

$$qe = \frac{(co-ce)}{m}.V \quad (2)$$

Where the quantities of adsorbed metal at the equilibrium are represented by the variables (q_t) and (q_e), respectively. The metal concentrations are (C_0, C_t, and C_e) (mg L^{-1}) at the beginning time, time t, an at the equilibrium time, respectively. V(L) denotes the volume of the metal solution, and m (g) is the adsorbent's weight.

In the experiment, the data obtained from the absorbed metal ion was fitted into Langmuir isotherm model using equation 3:

$$\frac{Ce}{qe} = \frac{1}{kL\ qmax} + \frac{ce}{q\ max} \quad (3)$$

where,

q_e (mg/g) = Equilibrium Adsorption Capacity

C_e (mg/L) = Equilibrium Adsorbate Concentration

qmax (mg/g) = Maximum Adsorption Capacity

K_L (L/mg) = Langmuir isotherm constant

Following that, in order to obtain Freundlich isotherms equation 4 was used [9]:

$$log\ qe = \log kf + \frac{1}{n}\log ce \quad (4)$$

where,

k_f = constant isotherm

n = adsorption intensity

Determined by plotting log q_e vs. log C_e, which gives a straight line with slope of 1/n and an intercept of log k_f. The magnitude of the 'n' shows an indication of the favourability of adsorption.

3. Results and Discussion

Preparation of the ligand was started by forming 3-methoxybenzoyl isothiocyanate through a nucleophilic substitution reaction. This was done by adding 3-methoxybenzoyl chloride to a stirring acetonitrile solution of ammonium thiocyanate (Figure 4). After filtration, the resulting solution was reacted with thiosemicarbazone in acetonitrile for four hours under refluxed. The colour of the solution changed into clear yellowish. The addition of salicylaldehyde immediately turned the solution into white cloudy solution. After two hours of reflux, and cooled to room temperature, the solution was poured into a conical flask that was soaked in a beaker containing some ice cubes. The precipitate was formed and filtered, then washed with little iced cold methanol. White precipitate was dried and kept for further investigation.

3.1 FTIR Analysis

Elucidation of the components in the ligands using FTIR, as shown in Figure 5, confirms the presence of crucial functioal groups. The N-H stretching vibrations in the range of 3270-3274 cm^{-1}, indicating the presence of imine group. The C=N stretching vibrations, found between 1684-1698cm^{-1}, suggest the formation of imine (Schiff base) linkage [6]. The aliphatic

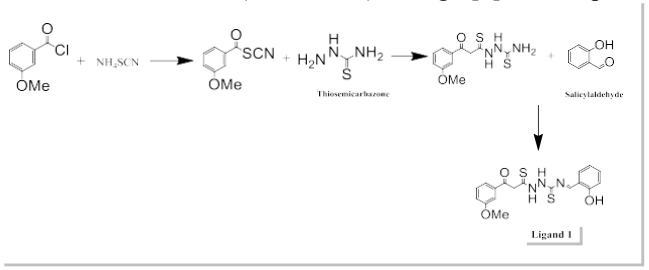

Fig. 4. Synthesis of N-(3 methoxybenzoyl)-N'-salicylidene thiosemicarbazone.

C-H represents methoxy appear around 2850-2950 cm^{-1}. The

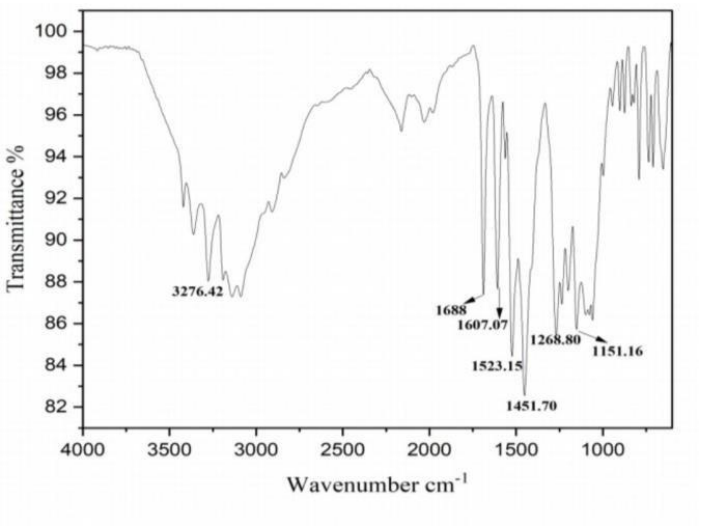

Fig. 5. FTIR spectra of the ligand.

C=C stretching vibrations in the region 1604-1610 cm^{-1} corresponding to aromatic rings, confirming the present of benzene moiety. The C=S bond vibrations, observed around 1271-1275 cm^{-1}, indicate of thiocarbonyl groups. This result validates the successful synthesis of the target compound and the expected functional groups [8].

3.2 NMR Analysis

The ^{1}H NMR spectrum of N'-(3-methoxybenzoyl)-N'-(salicylidene) thiosemicarbazone recorded in deuterated methanol (CD_3OD) displays key signals corresponding to various proton environments present in the compound, as shown in Figure 6. This analysis aims to assign and interpret each peak to validate the structure synthesized compound. The interpretation and structural confirmation are based on the formation of Schiff Base, the appearance of the singlet at 8.18ppm corresponding to the imine (-CH=N-) proton strongly confirms the successful formation of the Schiff base [4]. The chemical shift aligns with the imine protons adjacent to aromatic rings. Besides, aromatic region analysis shows the aromatic region between 7.4-8.0ppm displays distinct multiplets, doublets, and triplets, indicating various aromatic environments [10]. The splitting patterns and downfield shifts suggest conjugation with electron-withdrawing groups such as imine and carbonyl functionalities. Plus, thiosemicarbazone NH proton appear the singlet peak at 4.80ppm confirms the presence of an NH proton from the thiosemicarbazone structure. The slight broadness and downfield shift may result from hydrogen bonding or partial exchange with the solvent (CD_3OD) [11]. The signal at 3.32 ppm is a multiplet, which can be attributed to the O-CH_3 group on the 3-methoxybenzoyl ring. The chemical shift in this region aligns well with the expected position of a methoxy group on an aromatic ring. Its position is consistent with shielding by adjacent oxygen atom while maintaining slight deshielding from aromatic ring [12].

The ^{1}H NMR spectrum successfully supports the structure of ligand, validating the expected functional group, and aromatic protons confirms the synthesis. Thus, the spectral data aligns well with the proposed molecular structure.

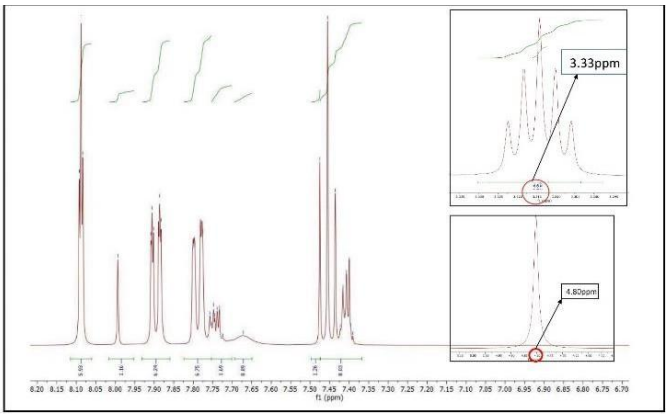

Fig.6. ^{1}H NMR spectra of the ligand

3.3 SEM-EDX Analysis

The impregnated of single-walled carbon nanotubes with ligand molecule was analysed using SEM-EDX. The SEM images, as shown in Figure 7, revealed the morphological changes occurring upon ligand attachment. The resulting sample has morphology of nanotubes were rope-liked, curved

and highly tangled and agglomerated [13]. The attachment of ligand into SWCNTs was shown at 15k magnification at range of 2-5 showing the white tangled strains on the surface of SWCNTs. These changes suggest successful functionalization of SWCNTs with respective ligand.

The observed morphological changes provide important evidence of the role of the hydrothermal-assisted retort treatment in facilitating ligand impregnation onto the SWCNTs surface. Under hydrothermal conditions, elevated temperature and autogenous pressure enhance solvent penetration into nanotube bundles, promoting partial bundling and increasing the accessibility of adsorption sites [14]. This process improves dispersion of SWCNTs and enables more uniform interaction between the ligand molecules and the nanotube surface.

The appearance of intertwined and coated nanotube structures in SEM images suggests that the ligand successfully anchored onto the SWCNTs framework. Surface modification is known to improve interfacial contact and generate additional active sites, which contribute to the enhance adsorption

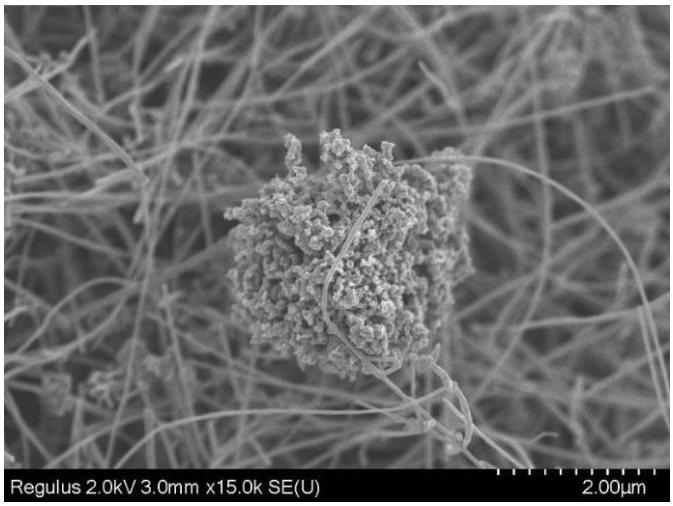

Fig. 7. SEM magnification of ligand onto SWCNTs

performance observed in this study [15]. The hydrothermal-assisted retort method plays a critical role in overcoming the common limitation of nanotube agglomerated and ensures effective functionalization compared to conventional solution impregnated techniques [16].

Elemental analysis using EDX, as shown in Figure 8, confirms the impregnation SWCNTs with the ligand. The presence of nitrogen (N) and sulphur (S), which are key elements in the ligand structures. Percentage of C element were 90.80%, N were 3.28%, O were 2.66% and S were 3.26%.

Hence, the SEM and elemental analysis confirm the successful impregnation of SWCNTs and the selected ligand with varying degrees of functionalization [17]. This suggests that ligand properties significantly influenced the extent of modification, which may impact the material's physicochemical behaviour and potential applications.

3.4 Adsorption isotherm

The Langmuir and Freundlich equation were used to describe the data derived from the adsorption of Co (II) by SWCNTs-ligand over the entire parameters range studied.

3.4.1 Langmuir Isotherm

The Langmuir isotherm assumes monolayer adsorption onto the surface with number of identical sites. The key parameters obtained from the Langmuir model include the maximum adsorption (q_{max}) and the Langmuir constant (K_L), which related to the affinity of the adsorbate for the adsorbent [13].

The Langmuir equation was used to fit the data and the adsorption parameter obtained such as maximum adsorption capacity, q_{max} (mg/g) and the Langmuir isotherm constant were shown in Figure 9 and Table 1.

Fig. 8. SEM-EDX spectrum of compounds present in modified ligand.

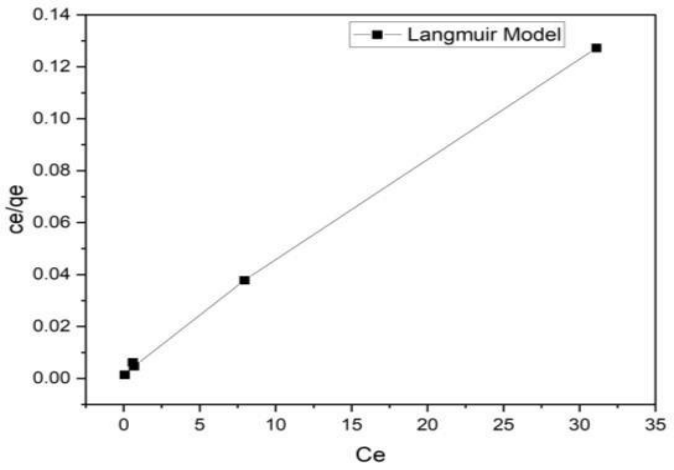

Fig. 9. Depicts the Langmuir plots of the adsorbed metal Co (II) on the impregnated SWCNTs with ligand.

Table 1. Adsorption parameters of metal Co(II) on the impregnated single-walled carbon nanotubes with ligand

q_e (mg/g)	q_{max} (mg/g)	K_L(mg/L)	R^2
244.45	248.75	1.34	0.99

From the Langmuir postulation, the metal ion was absorbed on the surface of the impregnated SWCNT with ligand. The R^2 values represent the coefficient of determination, which assessed the goodness of fit for the Langmuir model. The R^2 values the data range from 0.6774 to 0.9857, suggested a reasonably good fit for the Langmuir model [7]. The higher the R^2 values are, the closer they are unity or 1 which indicated an excellent fit between the experimental data and the Langmuir equation [3]. It was suggested at 80mg/L, the adsorption of the metal ion Co(II) excellently followed the Langmuir assumption. The determined value qmax of SWCNT-Ligand adsorbed on the Co (II) was 248.75 mg/g.

3.4.2 Freundlich Isotherm

The freundlich isotherm model describes adsorption on heterogeneous surfaces and accounts for multilayer adsorption. Figure 10 and Table 2 explained the Freundlich model's 1/nf values, which indicate surface heterogeneity or adsorption intensity, varied at 1 which is the favourite value of intensity. Some of metal ions Co (II) adsorbed onto the SWCNT- Ligand via the internal surface by particle diffusion, which is consistent with adsorption process took place. Generally, values of *n* between 1 and 2 indicate favourable adsorption conditions [9].

The finding show that metal ion Co(II) can effectively adsorb using the Langmuir and Freundlich isotherm models. The R^2 values for the Freundlich model range from 0.95 to 0.97, indicating a good fit. However, since the Langmuir model shows slightly higher R^2 values, it suggests that Co (II) adsorption onto these ligands is better described by monolayer adsorption rather than multilayer adsorption. Plus, from the SEM figure, the ligand attached at the surface of the SWCNTs which proven in Langmuir fitting parameters. Thus, the results indicate that Co(II) adsorption onto the ligand follows the Langmuir model more closely, implying monolayer adsorption on uniform sites [7].

Table 2. Adsorption parameters of SWCNT- Ligand on metal ion Co(II)

Slope	Intercept	n	Kf	R^2
0.7602	1.2099	1.315443	16.91517	0.9518

3.4.3 Effect on pH

The adsorption efficiency of cobalt removal was evaluated as a function of pH, as shown in figure, the graph illustrates the cobalt removal percentage for the sample across a pH range from 2 to 10.

From the results, it is evident that cobalt removal efficiency is significantly influenced by the pH 6. At lower pH values (2-3), the removal efficiency is relatively low, ranging from 10.24% to 15.32%. This may be attributed to the high concentration of hydrogen ions (H^+) competing with cobalt ions for adsorption sites, thereby limiting the adsorption of cobalt

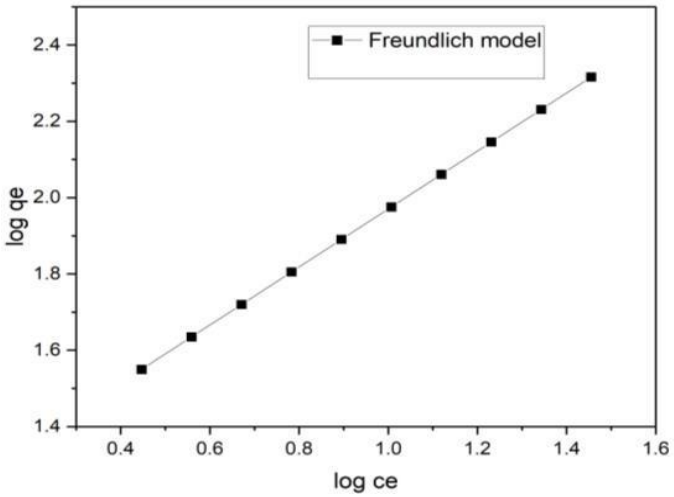

Fig. 10. Depicts the Freundlich plots of the adsorbed metal Co (II) on the impregnated SWCNTs with ligand.

onto the adsorbent [18].

As the pH increases, the cobalt removal efficiency improves, reaching its peak between pH 5 and 6, where the highest removal percentages are observed (30.21% and 35.01% for the most effective samples). This optimal range suggests that at moderately acidic conditions, the adsorbent surface exhibits favourable interactions with cobalt ions, leading to maximum adsorption. These findings indicate that pH plays a crucial role in determining the effectiveness of cobalt adsorption, with an optimal removal efficiency occurring around pH 6 which are valuable for adsorption process in practical applications [12].

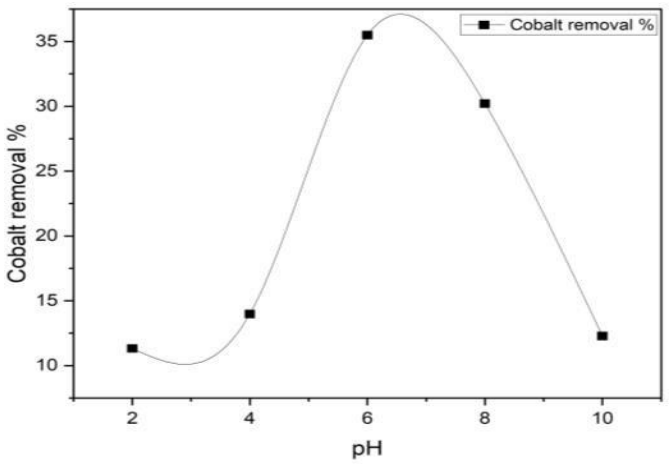

Fig.11. Removal percentages of Co(II) at different pH solutions

3.4.4 Effect on contact time

The graph depicts the variation of cobalt removal efficiency over contact time for the chelator. The trend suggests an initial rapid increase in cobalt removal, which then gradually stabilizes over time.

As shown in figure above, the effect of contact time on cobalt removal reach stability at 50 min range of time. In the first 15-35 minutes, cobalt removal increases rapidly, indicating that the adsorption process is fast at the beginning. This happens because, initially, there are many available adsorption sites in the material, allowing cobalt ions to easily attach to the surface. As more cobalt ions are adsorbed, the number of free sites decreases, and the process slows down [19]. After approximately 50 minutes, the removal efficiency stabilizes, meaning that equilibrium has been reached, most of the adsorption sites are occupied and not significantly improve cobalt removal.

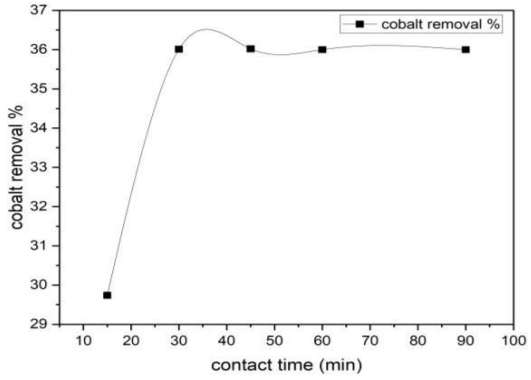

Fig.12. Removal percentages of Co(II) at different contact time.

the reaction follows a two-stage process which are a rapid initial phase where cobalt ions quickly attach to available sites, followed by slower phase where the remaining sites are harder to access, leading to a gradual plateau [6]. The results imply that the optimal contact time for cobalt removal is around 50 minutes, as extending the time further would have minimal effect on increasing removal efficiency. This behaviour is characteristic of adsorption-based removal systems, where the process follows a typical kinetic model [19].

3.5 Kinetic Adsorption

3.5.1 Pseudo-second order kinetic

Adsorption kinetic study was conducted to investigate chemical interactions between Co (II) ions and the adsorbent surface and showed the kinetic was follow pseudo-second order [12], [16]. The values of pseudo- second order rate constant k^2 and the adsorption capacity (q_e) are presented in table below.

Table 3. The parameter of the adsorption of Co(II).

Adsorbent (mg/L)	Qe (mg/g)	K^2(mg/min)	R^2
50	210.52	$4.27x10^{-2}$	0.99

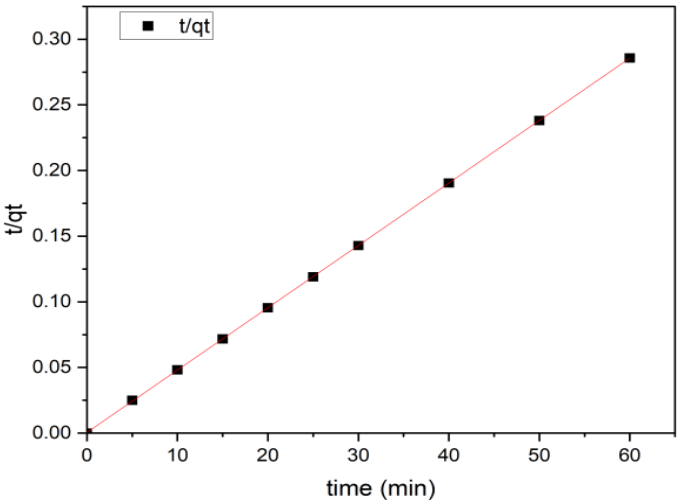

Fig.13. Pseudo-second order kinetic model for the adsorption of Co(II).

The plot of t/qt versus time as in figure above for the SWCNTs-ligand yields high correlation coefficients (R^2 = 0.99 (50mg). The second order-rate constant obtained from this figure is $4.27x10^{-2}$ mg/min. The second order-rate constant indicate that higher dosage of adsorbent required less time to achieve initial adsorption of Co (II) due to its potential surface area [5]. The equilibrium adsorption capacity, q_e obtained from the graph also implies that dosage of adsorbent (q_e = 210.52 mg/g) is capable to uptake more adsorbate. This result indicates that the rate controlling steps in the adsorption process is chemisorption, where interactions involved sharing or exchange of electrons between adsorbent and adsorbate [12]. The pseudo-second order kinetic model and the Langmuir isotherm from the kinetic study and the adsorption isotherm, respectively, support the involvement in the chemisorption in the adsorption process of SWCNTs-ligand [17].

4. CONCLUSION

In conclusion, the research on the impregnated single- wall carbon nanotubes (SWCNTs) with thiosemicarbone derivative s: Ligand N-(3-methoxy benzoyl)- N'(salicylidene)-thiosemi

carbazone has yielded significant results for metal ion Co (II) adsorption. The chemical characterization of the synthesis ligands reveals the presence of amide group and aliphatic compounds, particularly on thiosemicarbazone. The active functional group and significant molecular structure indicate its potential for further impregnated stage with single wall carbon nanotubes (CNTs) as an adsorbent for metal ion specifically Co (II). At mild pH around 6, the optimum efficiency for removal of the ion was observed in term of adsorption isotherms, using Langmuir and Freundlich adsorption isotherms models at five dissimilar initial concentrations. The data revealed that the Langmuir model, which suggests that Co (II) ion adsorb on both SWCNT- ligands' surface is the best model to represent the adsorption data with maximum adsorption capacity of248.75 mg/g. Respectively, the elimination of metal ion Co(II) at 80 mg/L for SWCNT-ligand was reported to be excel for adsorption study. Thus, impregnation of single wall carbon nanotubes (SWCNTs) with thiosemicarbazone derivatives N-(3-methoxy benzoyl)-N'(salicylidene)-thiosemicarbazone has ideal performance for adsorption of cobalt ion and applicable to be introduced in biomedical platform as drug delivery system for metal loaded disease.

ACKNOWLEDGEMENT

The authors would like to acknowledge the 600- RMC/GIP 5/3 (015/2023) from MARA University of Technology for financial support.

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