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RESEARCH

Synthesis and Characterization of Ni(II) Schiff Base Complexes onto 3-CPTMS/SBA-15

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ABSTRACT

Ni(II) Schiff base complexes containing diethylenetriamine-2,2'-bisphenol (L1) and 3,3'-iminebispropilamine-2,2'-bisphenol (L2) ligands were synthesized and embedded into SBA-15 functionalized with 3-chloropropyltrimethoxysilane (3-CPTMS/SBA-15). The characterization of the Ni(II) Schiff bases embedded into the mesoporous of 3-CPTMS-SBA-15 by elemental analysis, X-ray diffraction, nitrogen adsorption and desorption, and thermogravimetry, revealed that the mesoporous structures were maintained. From BET data, the surface area decreased from 517 m^2/g (SBA-15) to 326 m^2/g [Ni(L1)]-SBA-15 and 296 m^2/g [Ni(L2)]-SBA-15, with pore size diameter of ca. 5.6 nm. All materials presented isotherm type IV and H1 hysteresis. The TG/DTG curves showed the desorption of adsorbed water, coordinated water and ligands decomposition, and an increase in the thermal stability of the Ni(II) complexes embedded into SBA-15, evidencing that they are promising materials for adsorption, for remotion of heavy metals from aqueous media due to its chelating properties; and catalytic applications, because they contain oxygen and nitrogen as donor atoms, being of particular interest.

KEYWORDS

Nickel complexes, Functionalization, SBA-15, nanoporous material

INTRODUCTION

Schiff bases were prepared for the first time in the year 1864 by Hugo Schiff, with the condensation reaction between primary amine and carbonyl group (aldehyde or ketone). A Schiff base is a carbon-nitrogen double bond (-CH=N-) known as the azomethine group containing compound in which the carbonyl group (C=O) of an aldehyde or ketone has been replaced by primary amine. The azomethine group present in the Schiff base should play an important role for catalytic reactions. The nano-functionalized metal complexes of Schiff base ligands demonstrate the broad range of opportunities and challenges of this approach [**1,2**].

Schiff base complexes of metal ions show high catalytic activity and play a significant role in various reactions improving yield and product selectivity [**3-9**]. They are efficient catalysts for homogeneous and heterogeneous catalysis for reactions such as polymerization, oxidation, hydroxylation, aldol condensation and epoxidation. The activity varies with the metal ions and type of ligands in the coordination sites [**10**]. The complexes known as “salen” showed activity in ring opening oligomerization or polymerization of epoxides. The name “salen” is a contraction for salicylaldehyde and ethylenediamine. The Ni(II) Schiff base complexes of ethylenediimine and acetylaldimine were used for epoxidation of olefins with sodium hypochloride [**11**]. Aromatic Schiff bases or their metal complexes catalyze reactions on oxygenation [**12**], hydrolysis [**13**] and decomposition [**14**]. The anchoring of Ni(II) Schiff base into the MCM-41 via silicon alkoxide has been reported [**15,16**] Recent works report the application of Shiff bases in biomedicine [**17**], as catalyst for nitroaromatic reduction [**18**], and biological systems [**19**].

Mesoporous materials, such as silica, alumina and zeolite [**20**] have been evaluated as solid support for metal complex catalysts for applications in organic reactions. Since the discovery of the MCM-41 and SBA-15 materials in the 90's, these mesoporous materials have been extensively studied because of their appreciable characteristics such as high mechanical strength, thermal stability, porosity, surface area and presence of mesopore interconnected by micropores [**21-24**], making this solid as a great promise material for anchoring complexes with transition metals.

Schiff bases have attracted much attention in inorganic materials chemistry due to their ease of synthesis resulting from the condensation of primary amines with a compound having an active carbonyl group and because they form stable complexes when coordinated to transition metals with different states oxidation, thus, considered special ligands [**25**].

Transition metals Schiff bases encapsulated into mesoporous solids appear as new materials for biological and catalytic applications [**26**]. The use of nanoparticles and functional nanocatalysts, based on transition metal oxides, opens new opportunities for the application of more complex systems, such as those based on Schiff structures, which allow controlled release and specific functionalization in precision nanomedicine [**27**].

This work aims the functionalization of nanotubes of SBA-15 with 3-Chloro-Propyl-Tri-Methoxy-Silane (3-CPTMS) so that the square planar complexes of Schiff base [Ni(L1)], and [Ni(L2)] octahedral, where L1 = Schiff base using diethylenetriamine and L2 = Schiff base using the 3,3'-iminobispropilamina can be encapsulated into the SBA-15 mesoporous material. These reactants were used as coupling agents for surface modification of SBA-15.

EXPERIMENTAL

Synthesis of the Schiff bases

The synthesis of the Schiff base ligands was carried out as follows: first, 10 mmol of the carbonylated compound salicylaldehyde (Sigma-Aldrich, Saint Louis, MO, USA) were added to 60 mL of methanol (Merck Inc., Rahway, N.J., USA) under stirring and heated at 50 °C. Then, 5 mmol of diethylenetriamine (Sigma-Aldrich, Saint Louis, USA) was slowly added to the mixture and reacted for one hour. Then, for obtaining the [Ni(L1)] complex, 5.2 g of the $NiCl_2$.6H2O (Alfa Chemistry, NY, USA) and 5.2 g of sodium acetate were added, whereas for obtaining [Ni(L2)], the amine used was 3,3`-iminobispropylamine (with 1.2 g of nickel chloride. The obtained complex presented brown colour. Finally, the complexes were washed with cold ethanol (Merck Inc., Rahway, N.J., USA) and kept in the desiccators for five days.

Synthesis and functionalization of SBA-15

The SBA-15 was synthesized in strong acid medium, using tetraethyl orthosilicate – TEOS (Sigma-Aldrich, Saint Louis, MO, USA), as silica source and triblock copolymer P123 – EO20PO70EO20 (Sigma-Aldrich, Saint Louis, MO, USA) as template, according to hydrothermal procedure previously reported [**28**]. For the functionalization, 3.0 grams of the calcined SBA-15 mesoporous silica were suspended in 50 mL of 37% solution of HCl (Merck Inc., Rahway, N.J., USA) and stirred for one hour, washed with distilled water and dried in an oven for 24 h under vacuum. Then, the HCl-SBA-15 was refluxed with 2 mL of 3-Chloro-Propyl-Tri-Methoxy-Silane (3-CPTMS) (Merck Inc., Rahway, N.J., USA) in 40 mL of xylene (Merck Inc., Rahway, N.J., USA) for 24 h. The product was washed successively with ethanol, xylene, acetone and ethyl ether, and dried under vacuum at 100 ºC for 18 hours, to obtain 3-CPTMS/SBA-15.

Complex encapsulation in the pores of SBA-15

For encapsulation of the Ni(II) complexes, 1.0 g of 3-CPTMS/SBA-15 and 0.2 g of the [Ni(L1)] and [Ni(L2)] Schiff bases were left in suspension in 50 mL of toluene and refluxed for 24 hours. Then, the materials were recovered in a Soxhlet extractor at 60 ºC with toluene to remove the excess of the complex on the external surface of the SBA-15. The materials were dried in an oven for 30 min at 50 °C. After that, the solid was washed with water and ethanol to remove the complex which was not embedded and moved to an incubator at 50 °C for 24 hours. These functionalized materials were named as [Ni(L1)]-SBA-15 and [Ni(L2)]-SBA-15, where L1 is diethylenetriamine-2,2'-bisphenol and L2 is 3,3'-iminebispropilamine-2,2'-bisphenol ligands.

Materials characterization

The structural properties of the samples were analysed by Bruker model D2 Phaser diffractometer with CuKα source and a step size of 0.02°, in the range of 2θ = 1 to 10º. The nitrogen adsorption/desorption was performed with the materials, to obtain the BET surface properties, using equipment from Micromeritics Instrument Corporation TriStar II 3020 V1.03. For this analysis, the materials were previously degassed at 150 °C for 24 hours to remove the surface moisture and physiosorbed gases. The analysis was performed at -196 ºC at relative pressure (P/P_0) range of 0.01 to 0.95. The TG experiments were carried out using TG/DTG equipment model Q600, from TA Instruments, in the temperature range of 30 to 900°C, at heating rate of 10°C min^{-1}, with nitrogen gas flowing at 25 mL min^{-1}, using alumina crucible of 900µL.

RESULTS AND DISCUSSION

Chemical composition

According to the calculations of elemental analysis for C, H, N and Ni, the empirical formula for the compounds, were proposed, as summarized in **Table 1**, as well of the colour of them.

It was observed that the experimental values obtained for the complexes are consistent with theoretical values. Thus, the obtained results confirmed the molecular formulas and structures proposed for the complex, as $[Ni(L1)].2(CH_3CO_2).H_2O$, with L1 = $C_{18}H_{19}N_3O_2$ and $[Ni(L2)].2Cl.2H_2O$, with L2 = $C_{20}H_{23}N_3O_2$.

Table 1. Analytical and physical data for Ni(II) Schiff base complexes.

Empirical formula Proposed (Color)	Elemental analysis (%) Determined (Calculated)				Yield (%)
	C	H	N	Ni	
$[Ni(L1)].2(CH_3CO_2).H_2O$ (Orange)	52.4 (52.5)	5.3 (5.1)	8.3 (8.6)	4.4	53
$[Ni(L2)].2Cl.2H_2O$ (Green)	47.9 (47.6)	5.2 (5.5)	8.4 (8.9)	8.6	77

The empirical formula was proposed, considering the obtained values of elemental analysis for CHN.

Crystallographic properties

From X-ray diffraction of the materials, shown in **Fig. 1**, the hexagonal structure characteristic of mesoporous materials SBA-15 type were identified. Three major diffraction peaks were observed, concerning the crystallographic planes whose Miller indices are (100), (110) and (200), noting that even after anchoring the complex on the support, the SBA-15 mesoporous material did not lose their ordered hexagonal structure.

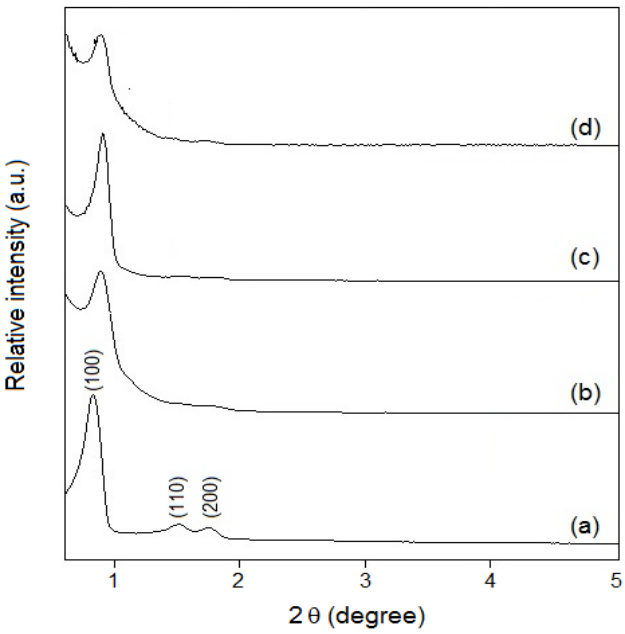

Fig. 1. XRD of the synthesized materials: (a) SBA-15; (b) 3-CPTMS-SBA-15; (c) [Ni(L1)]-SBA-15; (d) [Ni(L2)]-SBA-15.

Prior the functionalization process, the SBA-15 was treated with hydrochloric acid, in order to activate the surface with silanol groups (–Si–OH). It was suggested that these groups react with 3-CPTMS, and the tri-silanol species could be formed inside the mesopores of the SBA-15, as proposed in **Fig. 2**.

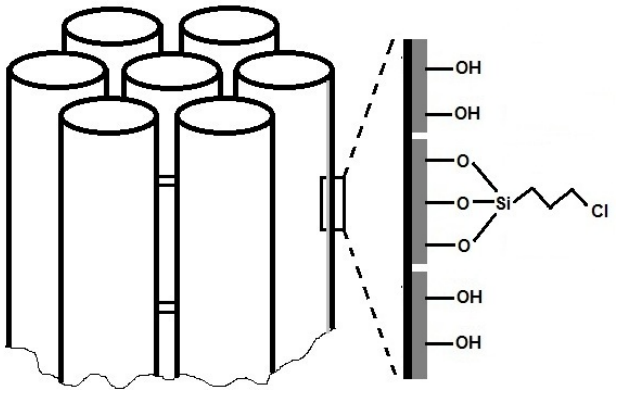

Fig. 2. Scheme proposed for functionalization of SBA-15 with 3-chloropropyltrimethoxysilane, for obtaining 3-CPTMS/SBA-15.

For SBA-15 functionalization with 3-CPTMS, it was necessary to activate the silanol groups, thus facilitating the bond between the compound and the silica structure. The success of the reaction depends on the availability of the silane groups to form covalent bonds with the silylating agent, which is possible due to the reactivity of the alkoxide groups with the silanol groups present on the surface of the silica SBA-15, which can occur tridentate form (see **Fig. 2**). Functionalization occurs so that there is greater efficiency in the anchoring of the complexes to silica.

The functionalized SBA-15 acts as starting material for reaction with the Ni(II)-Schiff bases. During the attachment of the Schiff base into the mesopore of the 3-CPTMS-SBA-15, the Ni(II) should bond covalently with the CPTMS. The proposed structures for [Ni(L1)]/SBA-15 and [Ni(L2)]/SBA-15 are shown in **Fig. 3**.

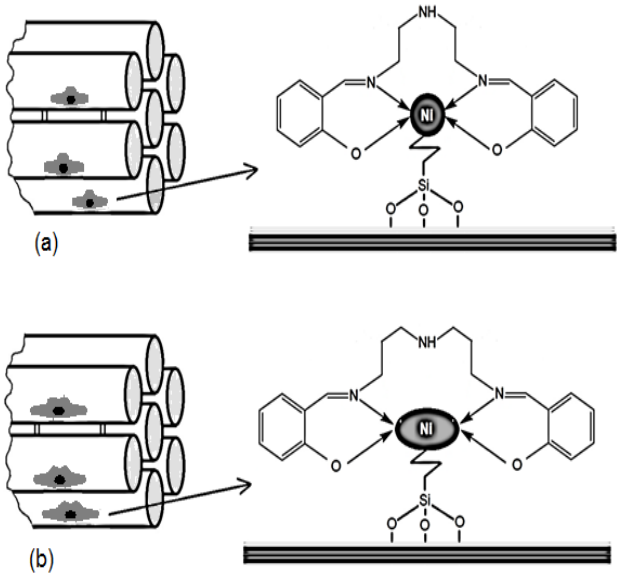

Fig. 3. Scheme proposed for encapsulation of Ni(II)-Schiff bases into the nanopores of SBA-15, where (a) [Ni(L1)]/SBA-15; (b) [Ni(L2)]/SBA-15.

Adsorption and desorption isotherms

The determination of the specific surface area (SA), pore diameter (Dp) and total pore volume (Vt) were obtained by the BET, BJH and t-plot methods, respectively [**29-31**]. The mesoporous parameter of the materials (a0) represents the distance between the pore centres of the SBA-15 structure, obtained from the (100) plane obtained from the X-ray diffractogram, and was calculated by applying Eq 01. These parameters are visualized in **Fig. 4**.

$$ao = \frac{2d(100)}{\sqrt{3}} \quad (01)$$

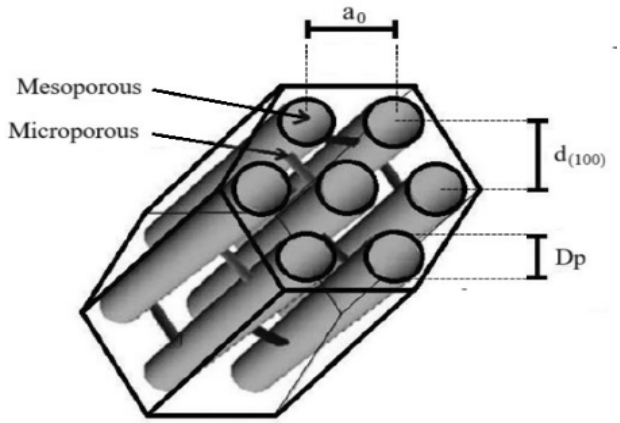

Fig. 4. Scheme for the hexagonal system for SBA-15 material, showing the representation of the mesoporous parameter, interlayer distance and pore diameter.

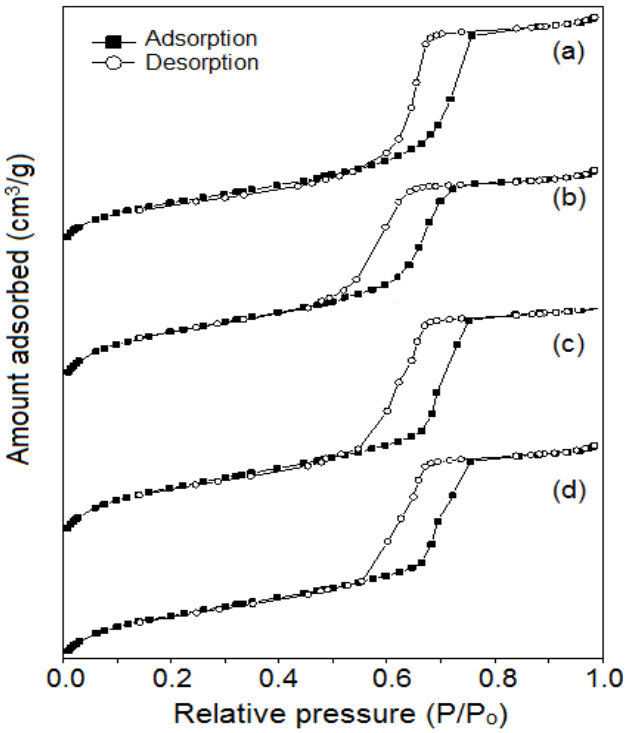

Fig. 5. Nitrogen adsorption and desorption isotherm for the obtained materials: (a) SBA-15; (b) 3-CPTMS/SBA-15; (c) [Ni(L1)]-SBA-15; (d) [Ni(L2)]-SBA-15.

Fig. 5 shows the N_2 adsorption and desorption properties in the materials. The surface area, diameter and volume of the material decreased during the changes in SBA-15 suggesting the presence of bulky material within the pores of the silica demonstrating a clear indication of the efficiency in functionalization process with 3-CPTMS for anchoring the complexes within the pores of the material system. The data relating to the adsorption properties for the obtained materials are given in **Table 2**.

Table 2. Textural properties of SBA-15, 3-CPTMS/SBA-15 and Ni(II) Schiff bases encapsulated in SBA-15 nanomaterial.

Sample	a_0 (nm)	SA (m^2/g)	Vp (cm^3/g)	Dp (nm)
SBA-15	12.4	517	0.74	6.18
3-CPTMS/SBA-15	11.3	334	0.46	5.08
[Ni(L1)]-SBA-15	11.4	326	0.50	5.62
[Ni(L2)]-SBA-15	11.5	296	0.45	5.61

a_0 = mesoporous parameter; SA = surface area; Vp = pore volume; Dp = pore diameter

Thermogravimetric analysis (TG/DTG)

The TG and DTG curves for obtained samples are shown in **Fig. 6**. Four ranges of temperatures were observed, corresponding to the following events: (i) adsorbed water; (ii) coordinated water; (iii) 3-CPTMS and (iv) ligands.

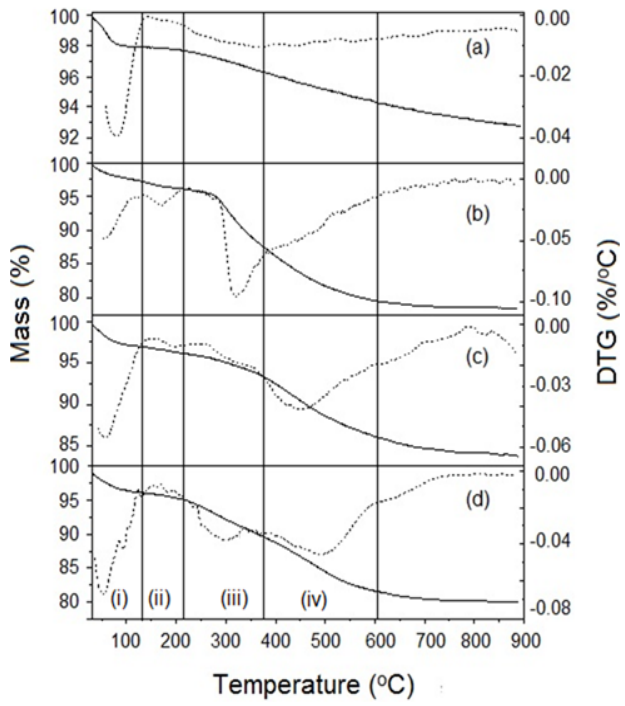

Fig. 6. TG and DTG curves for the obtained samples: (a) SBA-15; (b) 3-CPTMS/SBA-15; (c) [Ni(L1)]-SBA-15; (d) [Ni(L2)]-SBA-15.

The temperature range (i) occurs from room temperature up to 150 ºC, which corresponds to water adsorbed in the mesoporpous and microporous of the SBA-15. The coordinated water molecules were observed in the temperature range of 150-220 ºC. The calcined SBA-15 suffers slow dehydroxylation up temperatures higher than 600 ºC. The step (iii) corresponds to degradation of 3-CTPMS and it is clearly observed in the functionalized SBA-15, in the temperature range of 250-375 ºC. For the sample [Ni(L2)]-SBA-15, this DTG peak is observed, suggesting the presence of chloride interacting with the structure, as evidenced in the empirical formula of the Schiff base with L2 ligand. In the complexes encapsulated in the SBA-15, the removal of the organics from ligands were observed at maximum temperatures of 428 and 482 °C for [Ni(L1)]-SBA-15 and [Ni(L2)]-SBA-15, respectively, indicating an increased stability of the Schiff bases when encapsulated into the pores of SBA-15 mesoporous material.

CONCLUSIONS

Schiff bases complex of Ni(II) was successfully synthesized with a yield above 50%. The elemental analysis suggested a molecular formula for the complex: [Ni(L1)].2CH_3CO_2.H_2O and [Ni(L2)](2Cl).2H_2O (L1 = $C_{18}H_{19}N_3O_2$, L2 = $C_{20}H_{23}N_3O_2$). They were encapsulated inside the pores of SBA-15, and decreased the surface, however keeping the mesoporous parameter. From X-ray diffraction analysis, it was observed that even after anchoring the Schiff base complexes, the materials did not change its ordered hexagonal structure. The decrease in surface area, pore volume, pore diameter as compared to the SBA-15 confirmed the presence of complexes in the pores of the silica. By thermogravimetric analysis, it was noticed the greater thermal stability of the complex [Ni(L1)] and [Ni(L2)] when anchored in SBA-15. Schiff bases are a very important class of organic compounds because of their ability to form complexes with transition metal, such as Ni(II) ions for catalytic properties. Thus, metal complexes containing Schiff bases should be interesting compounds, because of its potential applications in designing new catalytic materials, especially when encapsulated in mesoporous structures.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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