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Synthesis, Characterization and Extractive Potentials of Aminopyrimidine Schiff Base Ligands on Divalent Metal Ions

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ABSTRACT

Two Schiff base (SB) ligands, HL and LH, consisting of both electron withdrawing and donating groups, derived from 2-hydroxyl-1-naphthaldehyde with 2-amino-4-hydroxy-6-methylpyrimidine and 2-amino-4-chloro-6-methylpyrimidine separately in the presence of CH_3CH_2OH and few drops of acetic acid at 40-60^oC was synthesized and examined for solubility and melting point. The multi-moiety ligand-molecules were recrystallized, acquired in good yields and further characterized using Infrared (IR) and ultraviolet (UV)-Visible spectroscopy. Extractive potentials of HL and LH were carefully investigated using atomic absorption spectroscopy (AAS) against the extraction of toxic metallic species: Ni^{2+}, Fe^{2+}, Zn^{2+}, and Co^{2+}, using a solvent-solvent extractive procedure. The synthesized HL and LH ligands exhibited highly selective extractive potentials towards Co^{2+} (99.9%), Zn^{2+} (99.7%) and Fe^{2+} (94.2%); and Ni^{2+} (59.25) separately. Acquired spectroscopic data revealed possible structural assemblages apportioned to the ligands.

Keywords: Aminopyrimidines, solvent extraction, divalent metal-ions, FT-IR, UV-Visible

INTRODUCTION

Solvent extraction popularly known as liquid–liquid extraction remains a procedure which permits the separation of twofold or more components owing to their imbalanced solubilities in different immiscible liquid phases [1]. It is one of the most adaptable procedures among the separation techniques often adopted for the removal and separation of metallic ions. Removal of metallic ions espousing organic molecules has remained an efficient technique in the extraction, separation, and/or pre-concentration of metallic species from the environment [3-4]. Solvent extraction has expansively been enhanced over the past four decades to become a frugally vital aspect of techniques utilized in the industry plus analytical chemistry research [5].

Solvent extraction has gained significance in analytical chemistry, petroleum, food and pharmaceutical industries in recent years as one method of high-efficiency and simplicity. This method is a quick and easy process as it also demands only very simple equipment [6]. The latter gained better relevance with SBs bearing multiple moieties. This is due to its characteristics and unique use in the extraction operations done on a large scale which increases the ability of the SBs to interact with many of the metallic ions since they contain chelating atoms [7].

Heavy metals remain toxic substances with their presence in the environment beyond certain concentration levels, thereby causing harm to known standard biological cycles. Industrialization (electroplating, mining and leather industries, pigments, metallurgical process, and textile industries) have occasioned in an upsurge in heavy metals contents in aqua bodies, triggering a problem that is yet to be entirely solved [8]. It is obligatory to remove as well as recuperate these extremely harmful plus non-biodegradable materials with the aim to acquire environmental quality standards, encourage the recycling as well as reuse of heavy metallic resources [9]. This has been done using countless methodologies including chemical precipitation, flocculation, filtration, chemical coagulation, reverse osmosis, ion exchange, solvent extraction, adsorption and membrane technologies [6]. Nonetheless, these approaches remain limited by high cost or may be insufficient in removal of heavy metal ions at trace level.

This paper consequently examines the synthesis of two novel SBs, HL and LH containing a pyrimidine moiety and evaluates their extractive potentials on selected divalent metallic ions. The ligands were characterized using spectroscopic techniques, melting point, solubility test, elemental analysis, etc to determine their structural assemblage while solvent extraction was adopted to establish their extractive potentials.

MATERIALS AND METHODS

All reagents; 2-hydroxyl-1-naphthaldehyde (2-HNA), 2-amino-4-chloro-6-methylpyrimidine (2-ACMP), 2-amino-4-hydroxyl-6-methylpyrimidine (2-AHMP), ethanol (CH_3CH_2OH), acetic acid, dichloromethane (CH_2Cl_2), chloroform ($CHCl_3$), dimethylsulfoxide ($(CH_3)_2SO$), dimethylformamide ($(CH_3)_2NCHO$), zinc(II)acetate dehydrate, nickel(II) acetate tetrahydrate, iron(II) sulphateheptahydrate, cobalt(II) chloride hexahydrate, and trietherlymine adopted were of analytical grade

Melting Point

Minimum amount of the SBs (HL and LH) properly isolated and stored in a clean dried bottle were introduced into a glass capillary tube with one end fused, inserted into a Digital Melting Point App. Machine and the melting points determined. The temperature at which the substance begins to melt were observed and recorded.

Solubility Test

The solubility of the SBs varied with the solvents used. Little amount of SBs were taken into a test tube and dissolved in series of solvents. The experiments were carried out with solvents like distilled H_2O, CH_3CH_2OH, $CHCl_3$, CH_2Cl_2, $((CH_3)_2NCHO)$ and $((CH_3)_2SO)$. In each case the different solubility exhibited by the SBs were also observed and recorded.

Preparation of Ligands

The SBs were prepared according to literature reported by Festus *et al.* [15] with negligible adjustments.

HL

To a hot ethanolic solution (40mL), 2-HNA (5g, 0.03mol) and 2-AHMP (3.75g, 0.03 mol) were added and constantly stirred carefully. The mixture was refluxed at 40-60 ˚C for 6 hr in the presence of few drops of acetic acid. On cooling, a pale yellow coloured compound was precipitated, filtered and washed with CH_3CH_2OH and dried.

LH

To a hot ethanolic solution (40 mL), 2-HNA (5g, 0.03mol) and 2-ACMP (4.30 g, 0.03 mol) were added and constantly stirred carefully. The mixture was refluxed at 40-60 ˚C for 6 hr in the presence of some drops of acetic acid. On cooling, a yellow coloured compound was precipitated, filtered and washed with CH_3CH_2OH and dried.

Solvent Extraction

Liquid-liquid extraction (Metal-Ligand)

The extraction of the metal ions using the synthesized SBs were carried out according to Morad *et al*., [10] with little adjustments. A 15 mL solution of $CHCl_3$ containing 7×10^{-5}M ligand and 15 mL aqueous solution containing 7×10^{-5} M divalent metal ions of Fe, Zn, Ni, Co each were prepared and mixed in a beaker which was placed on a magnetic stirrer. The mixture was stirred for 2 hr and the extraction was carried out at room temperature (27 ˚C) for LH. The aqueous and organic phases were separated and analysed using AAS with a solar thermo elemental flame. The extractability of the SBs was calculated using the equation below:

Extractability (%) = (M_O - M_A / M_O) × 100........... 1
Distribution ratio (D) = (M_O - M_A / M_O) 2

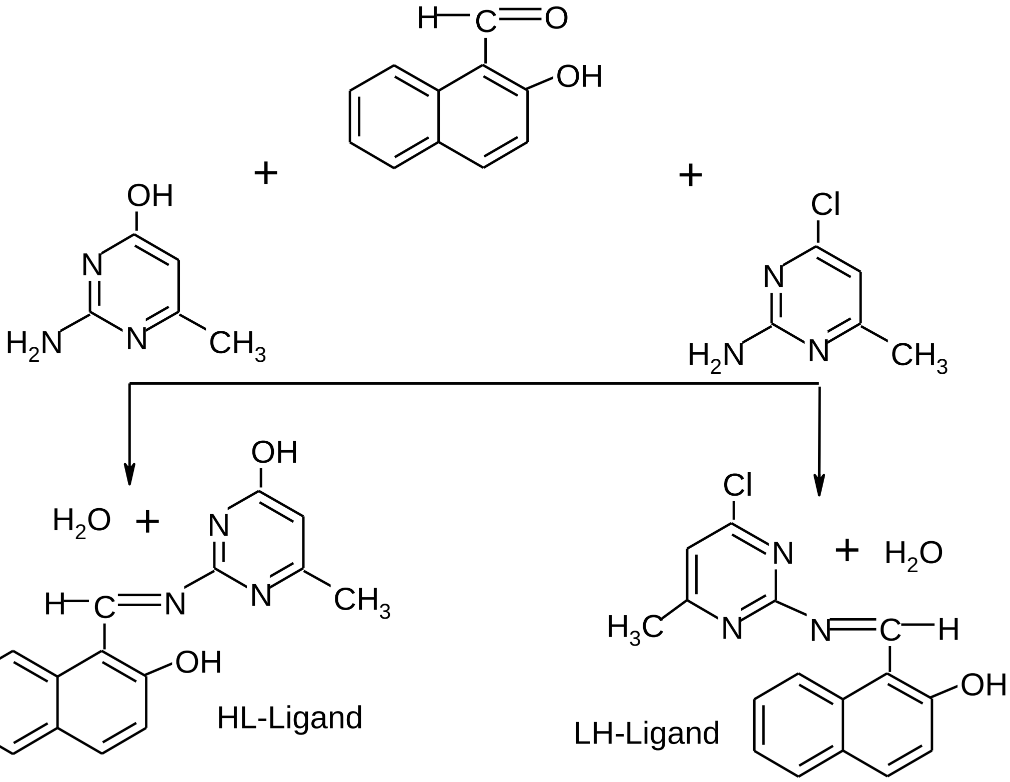
Where M_O and M_A = Concentration of metal ions in organic and aqueous phase separately.

Liquid-liquid extraction (Mixture of metals and ligand)

An organic solution 60 mL of the ligand in $CHCl_3$(7×10^{-5}M) was stirred for 2 hr with 60 ml of an aqueous solution (7×10^{-5} M) containing the mixture of metal ions (Fe, Zn, Ni, Co) on a magnetic stirrer. The extraction was carried out at room temperature (27˚C) for HL and LH. The aqueous and organic phases were separated and analysed using AAS with a solar thermo elemental flame. The extractability of the SBs was calculated using equation 1 above:

RESULTS AND DISCUSSION

Synthesis



Scheme 1: Synthetic Procedures for the SB Ligands

Analytical Results

The melting points, shades of colour and elemental analyses of the SBs are summarized in Table 1.

Table 1: Physical Characteristics and Analytical Data of the Ligands

Compound	Colour	Molecular formula	Molecular weight (g/mol)	Melting point (˚C)	Elemental analysis found (calc.)%				
					C	**H**	**N**	**O**	**Cl**
HL	Pale yellow	$C_{16}H_{13}N_3O_2$	280.307	100-114	68.80	4.69	15.04	11.45	-
LH	Yellow	$C_{16}H_{12}N_3OCl$	297.742	130-134	64.54	4.06	14.11	5.37	11.90

Solubility Test

The solubility tests in Table 2, displays the common behaviour of the ligands, HL and LH exhibited in some of the selected solvents.

Table 2: Solubility test of the ligands

Ligands	Solvents used					
	$(CH_3)_2SO$	$(CH_3)_2NCHO$	CH_2Cl_2	H_2O	CH_3CH_2OH	$CHCl_3$
HL	S	SS	SS	IN	SS	S
LH	S	S	S	IN	S	S

Key: S-Soluble, SS-Slightly Soluble, IN-Insoluble

Extraction Studies

The results of analysis for the ligands, HL and LH are presented in Tables 3 and 4 respectively, and represented on a chart as shown in Figure 1.

Table 3: % Extraction of Selected Metal ions using HL synthesized ligand

Metal ions	Aqueous phase	Organic phase	Distribution ratio	% Extraction
Zn^{2+}	0.045	18.043	0.9975	99.75
Fe^{2+}	1.332	23.356	0.9429	94.29
Ni^{2+}	121.039	135.550	0.1070	10.70
Co^{2+}	<0.001	39.526	0.9999	99.99

Key: Aqueous phase (metal ions) = 1×10^{-7}M; Organic phase (ligands)=1×10^{-7}M; at 25℃ for 2hrs contact time.

Table 4: % Extraction of selected metal ions using LH synthesized ligand

Metal ions	Aqueous phase	Organic phase	Distribution ratio	% Extraction
Zn^{2+}	0.146	0.148	0.0135	1.35
Fe^{2+}	137.960	116.317	0.1568	15.68
Ni^{2+}	127.322	312.482	0.5925	59.25
Co^{2+}	49.115	50.091	0.0194	1.94

Key: Aqueous phase (metal ions) = 1×10^{-7}M; Organic phase (ligands) = 1×10^{-7}M; at 25˚c for 2hrs contact time.

Table 5: FTIR Frequencies (cm^{-1}) of the SBs

Ligands	$^{\gamma}$C-C	$^{\gamma}$C=C	$^{\gamma}$C=O	$^{\gamma}$C-N	$^{\gamma}$N-H	$^{\gamma}$C=N	$^{\gamma}$OH	$^{\gamma}$C-H	$^{\gamma}$C-O	$^{\gamma}$C-Cl
HL	1470	1651	-	1315	-	1667	3334	3076	1248	-
LH	1035	1645	-	1128	-	1683	3635	2919	1365	778

Table 6: UV-Visible Spectra of the ligands

Ligands		UV-Vis Spectra					
	λ_{max} (nm)	193	254	296	322	373	378
HL	λ_{max} (cm^{-1})	51813	39370	33783	31055	26809	26455
LH	λ_{max} (nm)	250	295	315	330	340	375
	λ_{max} (cm^{-1})	40000	33898	31746	30303	29411	26666

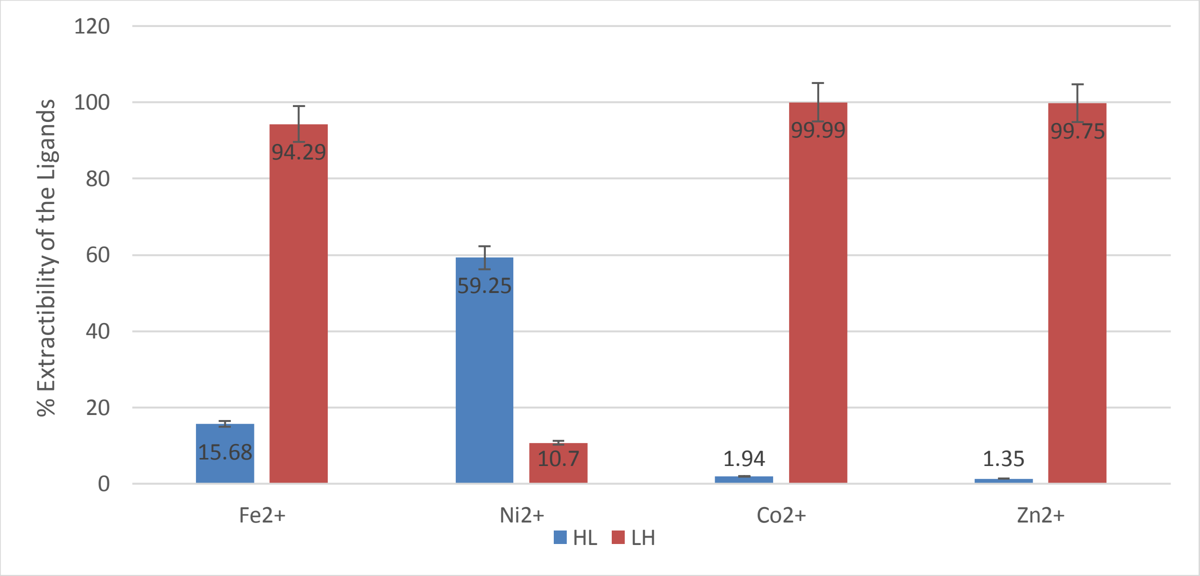


Figure 1: Graphical representation of the % extraction of the selected metallic ions using the synthesized HL and LH ligands.

Analytical Analysis

The physical characteristics and analytical data of the ligands HL and LH reported in Table 1 consists of the proposed chemical compositions of the ligands. It also revealed the coordination of the ligands in a ratio of 1:1 stoichiometry. The melting points observed were sharp indicating the purity of the synthesized SB ligands [11].

Solubility Test of the Ligands

The ligands were observed to be soluble in $(CH_3)_2SO$ probably due to its high dielectric constant, and slightly soluble in $(CH_3)_2NCHO$, CH_2Cl_2, CH_3CH_2OH and $CHCl_3$. However, the ligands were insoluble in H_2O as shown in Table 2 [12].

Extractive potential of the ligands

Tables 3 and 4 summarised the percentage (%) extraction of the metallic ions (Zn^{2+}, Fe^{2+}, Ni^{2+}, Co^{2+}) using the synthesized ligands (HL and LH). The % extraction of Zn^{2+}, Fe^{2+}, Ni^{2+}, and Co^{2+}ions using HL exhibited 99.7%, 94.2%, 10.7%, and 99.9%; while LH gave 1.3%, 15.6%, 59.2% and 1.9% singly. The results indicate that the % extraction of metallic ions increases in the order $Co^{2+}>Zn^{2+}>Fe^{2+}>Ni^{2+}$ and $Ni^{2+}>Fe^{2+}>Co^{2+}>Zn^{2+}$ for HL and LH separately.

In Table 3, the percentage (%) extraction of the selected metallic ions using HL synthesized ligand showed that the ligand exhibited a good to excellent extractive potential. The ligand efficiently extracted Co^{2+} (99.9%), Zn^{2+} (99.7%) and Fe^{2+} (94.2%) ions at 27˚C from the aqueous phase into the organic phase. However, the C=N functional group bound to the imine group increases the extractability of the ligand because the donor structure of the nitrogen atoms causes a very good to excellent extractive potential of HL ligand towards the metallic ions [13]. It was also observed that the HL ligand had a poor extractive potential towards the Ni^{2+} ion which could be due to the presence of a lower electronegative OH group attached to the HL ligand.

The results of Table 4 denote the percentage (%) extraction of the selected metallic ions using the synthesized LH ligand which revealed that the ligand exhibited a good to very poor extractive potential. The metallic ion Ni^{2+} (59.25) gave a relatively good extractive potential. This probably is as a result of the high electronegative atom (Cl) attached to the ligand LH. As collated to the extractability of the Ni^{2+} ion, the ligand poorly extracted the metallic ions Zn^{2+} (1.3%), Fe^{2+} (15.6%) and Co^{2+} (1.9%) from the aqueous phase into the organic phase probably due to individual extraction of the metallic ions which causes a decrease in the affinity of the synthesized LH ligand towards the metallic ions [10].

From Tables 3 and 4, the percentage (%) extractive potential of the synthesized ligands deduced were in the order HL>LH. It was also observed that the extractive potentials of the synthesized HL ligand was higher compared to the extractive potential of the synthesized LH ligand probably due to the individual extraction of the metal ions as compared to that of the mixture of metal ions to ligands. Generally, the HL ligand displayed an exceptional selectivity for $Zn^{2+,}$ Fe^{2+}, and Co^{2+}while LH exhibited relatively good selectivity towards Ni^{2+} ion.

Infrared Spectra

The IR spectrum gives significant information about the nature of functional groups involved in coordination mostly in synthesized organic compounds. The spectra of the ligands synthesized and reported for this work were obtained over a spectral range of 4000-450 cm^{-1} as KBr disc. The vital FT-IR spectral data of the ligands are presented in Table 5 and were associated to documented literature reports [14-15] on discussion and assignment of the bands.

The presence of the strong band observed at 3334cm^{-1} was assigned to ʸO-H within the HL ligand assemblage. The latter corroborates the existence of a hydroxyl functional group typical of SBs derived from 2-hydroxyl-1-napthaldehyde [2]. The appearance of the band at 1667cm^{-1} assigned to ʸC=N showed that the free primary NH_2 moiety of amino-pyrimidine group was converted to azomethine group [16]. The spectrum also showed ʸC-H stretching bands at 3076cm^{-1} due to the aromatic rings contained within the synthesized HL ligand [17-18]. The intensity bands observed at 1470cm^{-1}, 1651cm^{-1},1315cm^{-1} and 1248cm^{-1} were assigned to ʸC-C, ʸC=C, ʸC-N, and ʸC-O individually.

Within the IR spectrum of the ligand LH, the characteristic band at 3635 cm^{-1} was assigned to ʸO-H confirming the presence of the hydroxyl functional group of naphtha-aldehyde on the SB [16] and an amine (ʸC=N) band appeared at 1683cm^{-1}stretching vibration signifying the formation of the SB [19]. The stretching vibration at 2919cm^{-1} was assigned to ʸC-H as a result of the methyl group attached to the pyrimidine moiety of the synthesized ligand [18, 20]. Accordingly, the bands observed at 1309 cm^{-1}, 1645 cm^{-1}, 1128 cm^{-1}, 1365 cm^{-1} and 778 cm^{-1} were apportioned to ʸC-C, ʸC=C, ʸC-N, ʸC-O and ʸC-Cl separately.

UV-Visible spectra

The absorption region assignments of the ligands are given in Table 7 and were obtained at the range of 19000-60000 cm^{-1}. The HL synthesized ligand had peaks within the ultra-violet spectra at the range of 26455 cm^{-1} -26809 cm^{-1}, 31055 cm^{-1} -39370 cm^{-1} and 51813 cm^{-1} allocated to n-π*, π- π* [21] and charge transfer transitions singly. The transitions observed in the synthesized HL ligand indicated the presence of different functional groups on the SB; π→π*(ʸC=C), n→π*(ʸC=N) [22] ʸO-H(n→σ*)ʸC-O,ʸC-N and ʸC-C, ʸC-H (σ→σ*) individually [23].

The ultraviolet (UV) spectrum of LH ligand displayed intra-ligand transitions n→π* with absorption bands of 26666 cm^{-1}– 29411 cm^{-1}, π- π* transition with absorption bands of

30303– 33898 $cm^{-1}$and absorption band of 40000 cm^{-1} observed were also assigned to n→π* transitions [24] singly. The ultra-violet spectra had transitions attributed to different functional groups; π→ π* (ɣC=C), n→π*(ɣC=N,ɣO-H), n→σ*(ɣC-O,ɣC-N,ɣC-Cl) and σ→σ*(ɣC-C, ɣC-H) individually [23].

CONCLUSION

In this research work, the synthesized and characterized HL and LH ligands derived from the reflux condensation reaction of 2-HNA in addition to2-AHMP and 2-ACMP respectively were in good agreement with the solubility test, elemental analysis, melting point, IR and UV-Vis studies reported by other literatures. The extractive potentials of the synthesized ligands HL and LH were highly selective towards Co^{2+}, Zn^{2+}; and Fe^{2+} and Ni^{2+}separately.

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