Chemistry of platinum and iridium complexes of thioethers: contributions of P. C. Ray and related later developments

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Manuscript received 27 October 2014, accepted 31 October 2014

Abstract: The platinum species studied include planar \([\text{Pt}^{II}(\text{R}_2\text{S})_2\text{Cl}_2]\) and octahedral \([\text{Pt}^{IV}(\text{R}_2\text{S})_2\text{Cl}_4]\) both of which occur in cis and trans isomeric forms. Ray’s work concerned their synthesis (from \(\text{H}_2\text{Pt}^{IV}\text{Cl}_6\) and \(\text{R}_2\text{S}, \text{R} = \text{Et, also Me, CH}_2\text{Ph}\)) and reactivity towards amine ligands as well as assignment of isomeric structure. Others including Werner had worked in this area prior to Ray. However the correct assignment of isomeric structure could only be settled later by others through application of physical tools. Ray also examined \([\text{PtCl}_3\cdot 2\text{R}_2\text{S}]\) which he correctly identified as a mixed valence compound which he formulated as a loose 1:1 combination of \([\text{Pt}^{II}(\text{R}_2\text{S})_2\text{Cl}_2]\) and \([\text{Pt}^{IV}(\text{R}_2\text{S})_2\text{Cl}_4]\). However, it is more likely that it has the ionic structure \([\text{Pt}^{II}(\text{R}_2\text{S})_4\text{Cl}_2]\)[\text{Pt}^{IV}\text{Cl}_6]\) proposed earlier by Tschugaeff. The first-ever thioether coordination chemistry of iridium originated from Ray’s laboratory. By reacting \(\text{IrCl}_4\) with \(\text{R}_2\text{S}\) he isolated two (yellow and red) isomeric compounds of formula \([\text{Ir}^{III}(\text{R}_2\text{S})_3\text{Cl}_3]\) and studied their reactions with pyridine. He assigned fac and mer geometries to the yellow and red forms respectively. However physical tools revealed decades later that the yellow isomer actually has mer geometry and the red isomer is an ionic dimerization isomer \([\text{Ir}^{IV}(\text{R}_2\text{S})_2\text{Cl}_2]\)[\text{Ir}^{IV}(\text{R}_2\text{S})_2\text{Cl}_4]. The \([\text{Ir}^{III}\text{Cl}_3(\text{R}_2\text{S})_3]\) system of Ray was the first member of the general class \([\text{M}^{III}\text{X}_3(\text{R}_2\text{S})_3]\) (M is 4d or 5d metal and X is Cl or Br) discovered by others later. All appear to have mer geometry.

Keywords: Thioether complexes, platinum (bi-, tetra- and mixed-valent), trivalent iridium, geometrical isomerism, dimerization isomerism.
Physicochemical, computational and DNA cleavage studies of $N'\prime\prime\prime\prime-[(1E)-1-(2,4-dihydroxyphenyl)ethylidene]benzenesulfonohydrazide and its metal complexes

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Manuscript received online 05 June 2014, revised 10 July 2014, accepted 22 July 2014

Abstract: Benzenesulfonohydrazides and their metal complexes are important compounds in medicinal chemistry. In the present investigation synthesis of $N'\prime\prime\prime\prime-[(1E)-1-(2,4-dihydroxyphenyl)ethylidene]benzenesulfonohydrazide, its metal complexes and their structural properties have been investigated by various spectro-analytical and computational studies. Molecular orbital and Quantitative Structure Activity Relationship (QSAR) properties were computed by employing HyperChem 7.5 tools. The pH-metric studies indicated dibasic nature of compound with two corresponding pKa values. The pKa values were further compared with computed values derived by using ChemAxon tools. The interaction of metal ions with candidate compound was monitored by Job’s method of continuous variation and mole-ratio methods in ethanol-water medium. The solid metal complexes synthesized were investigated by various techniques viz. IR, UV-Visible, mass, ESR, SEM, TG, DTA and elemental analysis. The DNA cleavage studies carried out on the title compound and its metal complexes indicated that only copper complex has the property to cleave the super coiled DNA to linear form.

Keywords : Sulfonohydrazide [{(N-dihpesh)-H$_2$}], computational studies, equilibrium studies, DNA cleavage.
Abstract: Two dinuclear mercury(II) halide complexes of the type [(L)Hg(µ-X)HgX₃] (L = tris(2-aminoethyl)amine, 1; X = Cl; 2; X = Br) have been synthesized and X-ray crystallographically characterized. Structural analyses show that 1 and 2 contain two different halide bridged mercury(II) centers, with Hg₅X₄ distorted trigonal bipyramidal and Hg₅X₄ tetrahedral chromophores, respectively. Weak intermolecular N–H···Cl/Br and C–H···Cl/Br hydrogen bonds in 1 and 2 promote dimensionalities. The compounds show reasonable thermal stability.

Keywords: Mercury(II) halide, dinuclear compound, tripodal amine, X-ray structure, thermal behavior.

Syntheses of (E)-4-(2-(4-methoxyphenyl)diazemyl)benzene 1,3-diol and (E)-1-(2-(4-hydroxyphenyl)diazemyl)-2-naphthol and their application as acid base indicator and in jute dyeing

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Manuscript received 22 January 2014, accepted 31 July 2014

Abstract: Two novel azo-dyes namely (E)-4-(2-(4-methoxyphenyl)diazemyl)benzene 1,3-diol (L¹) and (E)-1-(2-(4-hydroxyphenyl)diazemyl)-2-naphthol (L²) have been synthesized by the usual method of diazotisation followed by coupling reaction at pH 7.5. The synthesized dyes are characterized by elemental analysis and UV-Vis, IR, ¹H NMR, mass spectrometry. These synthesised compounds are found to exhibit significant dyeing property on bleached jute substrate under neutral and particularly basic pH condition with good colour yield and wash fastness. Moreover the acid base indicator property of L² being more prominent than that of L¹, the parameters measuring the performance of the former as an indicator have also been evaluated spectrophotometrically and conductometrically in this paper. The study shows better performance of L² than methyl orange, particularly for titration of dilute solutions.

The antibacterial activity of both L¹ and L² has also been studied, where L² showed more significant effect than L¹.

Keywords: Azo-dye, jute dyeing, acid-base indicator, antibacterial effect.

FTIR, ¹H NMR, mass spectral, XRD and thermal characterization studies of NdIII and SmIII complexes of glipizide: An oral antidiabetic drug

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Manuscript received online 10 May 2014, revised 14 July 2014, accepted 04 August 2014

Abstract: The present paper deals with the study of sulphonylurea glipizide (GLP) drug in order to give a thought concerning its coordinating potentiality towards some inner transition metals. Metal complexes of glipizide drug is prepared and characterized based on analytical data, molar conductance, IR, ¹H NMR, mass spectrometry, X-ray diffraction and thermogravimetric analysis.
(TGA) studies. From the analytical data, the complexes are proposed to have general formulae \((\text{C}_2\text{H}_2\text{N}_2\text{O}_3\text{S}_1\text{Nd(OH)}_2)_2\) and \((\text{C}_2\text{H}_2\text{N}_2\text{O}_3\text{S}_1\text{Sm(OH)}_2)_2\). Low values of molar conductance indicate that complexes have non ionic nature. The conductometric titration using monovariation method reveal that complexes are L\(_2\)M type which is further confirmed by Job's method of continuous variation as modified by Turner and Anderson. Geometry of complexes are assigned to be hexagonal in which ligand molecules lie horizontally joining the central metal atom and four water molecules attached vertically and horizontally with the metal, supported by IR, \(^1\)H NMR and mass studies. Powder X-ray diffraction data have been used to calculate particle size, porosity, volume of unit cell and density of synthesized complexes. The thermal decomposition of complexes is studied using thermogravimetric (TGA) techniques. The thermal parameters such as, energy of activation \((E_a)\), enthalpy \((\Delta H)\), entropy \((\Delta S)\) and free energy change \((\Delta G)\) of the complexes is evaluated by employing the Freeman-Carroll and Sharp-Wellworth methods and the relative thermal stability of the complexes are discussed.

Keywords : Glipizide, antidiabetic drug, metal complexes, spectroscopy, XRD, TGA.

J. Indian Chem. Soc.,
Vol. 92, January 2015, pp. 65-69

**Extraction studies of Zn\(^{II}\) from salicylate medium using Cyanex-923 extractant in toluene**

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*Manuscript received online 29 May 2014, revised 04 August 2014, accepted 07 August 2014

**Abstract** : Cyanex-923 extractant has been used for the extraction of Zn\(^{II}\) from sodium salicylate medium. This metal ion was quantitatively extracted with Cyanex-923 in toluene at pH 5 and from the organic phase it is stripped back with 1 M HCl solution. The effect of pH, sodium salicylate concentration, reagent concentration, equilibration period, diluents, diverse ions and stripping agent on the extraction of Zn\(^{II}\) has been studied. The stoichiometry of the extracted species was determined on the basis of slope analysis method. The extraction proceed by solvation mechanism and the probable extracted species was Zn(Sal)\(_2\).2Cyanex-923.

Keywords : Zn\(^{II}\), Cyanex-923, toluene, multicomponent mixtures, nycil powder.

J. Indian Chem. Soc.,
Vol. 92, January 2015, pp. 71-77

**Synthesis, spectral characterization and DNA binding properties of dinuclear copper(II) complexes with pyridinehydrazones**

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*Manuscript received online 22 March 2014, revised 21 June 2014, accepted 10 August 2014

**Abstract** : Dinuclear copper(II) complexes of functionalized heterocyclic hydrazones, viz. 2-acetylpyridine acetoylhydrazone (APAH), 2-acetylpyridine benzoylhydrazone (APBH), 2-benzoylpyridine acetoylhydrazone (BPBH), 2-benzoylpyridine benzoylhydrazone (BPRBH) have been synthesized and characterized on the basis of elemental analysis, molar conductivity measurements, magnetic susceptibility, electronic, infrared and electron spin resonance spectral data. Electrolytic nature of complexes is investigated by conductivity studies. IR spectral data suggest that the ligands act as neutral trifunctional NNO-donor system. Electronic spectral data suggest octahedral geometry for the complexes. Electrochemical behavior of metal complexes is investigated by using cyclic voltammetry. The complexes undergo quasi-reversible one electron reduction. Absorption titration studies revealed that these complexes are avid binder to calf-thymus DNA. The electronic absorption
titrations suggest that the complexes bind DNA through intercalation involving a strong π-stacking interaction between the aromatic chromophore and base pairs of DNA.

Keywords: Copper complexes, pyridine hydrazones, synthesis, spectral studies, DNA binding properties.

J. Indian Chem. Soc.,
Vol. 92, January 2015, pp. 79-88

Synthesis, structure, electrochemistry, fluorescent and DFT study of Ru\textsuperscript{II} complexes with pincer-type 2,6-bis-(N-methylimidazolylidene/benzimidazolylidene)pyrazine ligands

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Abstract : Pyrazine based CNC-pincer proligands 2,6-di-(N-methylimidazolium)pyrazine dichloride, 1; 2,6-di-(N-methylbenzimidazolium)pyrazine dichloride, 2 and their Ru\textsuperscript{II}-N-heterocyclic carbene (NHC) complexes bis-[2,6-di-(N-methylimidazo-2-ylidene)pyrazine]ruthenium(t) hexafluorophosphate, 3 and bis-[2,6-di-(N-methylbenzimidazo-2-ylidene)pyrazine]ruthenium(t) hexafluorophosphate, 4 have been synthesized and characterized by different spectroscopic techniques. Electrochemistry and emission spectra of the compounds have been studied. The voltammetric feature of complexes 3 and 4 are quasi-reversible to irreversible as it is evident from large $\Delta E_p$ data ($>$250 mV). Both complexes are fluorescence active in nature.

Keywords : N-Heterocyclic carbene, ruthenium(t), CNC-pincer, electrochemistry, spectra, DFT.

Spectrophotometric studies on the charge-transfer complexes between tetryl with amines in DMSO and determination of the vertical electron affinity of tetryl

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Abstract : Tetryl (N-2,4,6-tetranitro-N-methylaniline) formed beautifully colored complexes with aliphatic amines like isopropylamine, ethylenediamine, tetraethylenepentamine and bis(3-amino propyl)amine acting as chromogenic agents in DMSO having two distinct peaks near 447 nm and 517 nm respectively. The complexes were stable for 12 h. The extinction coefficients (fairly high)
of the amino complexes of tetryl were determined directly by using the concentration of amines 400 times more than that of tetryl. The complexes were observed to be 1 : 1 type using Job’s method of continuous variations. The thermodynamic association constants of the complexes were determined directly using the extinction coefficient values at 298 K at two wavelengths. The results agreed well. The results were also verified by calculating $K_{DA}$ using the iteration method. The energies of transition for the CT complexes $h\nu_{CT}$ found experimentally agreed reasonably well with the theoretical energies of CT transitions (from HOMO to donor and LUMO to acceptor) obtained using DFT calculations. The vertical electron affinity $E_{AV}$ of tetryl was calculated using the method suggested by Mulliken. However, FTIR measurements of the tetryl complexes could not performed due to experimental and technical limitations.

Keywords : Association constants ($K_{DA}$), CT complexes, DMSO, spectrophotometry, vertical electron affinity.

J. Indian Chem. Soc., Vol. 92, January 2015, pp. 97-103

Investigation on heavy metal content in common grown vegetables from polluted sites of Moradabad district, India

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Manuscript received online 01 June 2013, revised 29 July 2014, accepted 08 August 2014

Abstract : Vegetables growing on heavy metal contaminated soils can accumulate high concentrations of trace elements and may pose serious health risk to consumers. Essential trace elements (Fe, Zn, Mn, Cr) were analyzed in edible and non-edible parts of six vegetables i.e. palak, bathua, shalgam, cauliflower, cabbage and coriander collected from Dhaurina and Karula nala at Moradabad district using AAS. Concentration of Cr in all vegetables and Zn in Dhauri palak roots and leaves, Karula nala bathua roots and leaves and Karula nala shalgam crossed the tolerable limits for human consumption. Transfer factor of Cr was observed to be maximum. There was risk of consumption of examined vegetables, as daily intake for chromium was above the tolerable daily intake value.

Keywords : Heavy metals, daily intake, transfer factor, vegetables, polluted site.


Comparative phytochemical analysis of wild and cultivated rhizomes of Hedychium spicatum Buch. Ham. of north west Himalaya


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Manuscript received online 02 November 2013, revised 29 July 2013, accepted 08 August 2014

Abstract : The pharmaceutical, cosmetic and agricultural demand of medicinal and aromatic plant species causes promotion of their commercial cultivation in order to meet the increasing demand within the domestic and export markets. Rhizomes of Hedychium spicatum Buch. Ham. (Family : Zingiberaceae), contain bioactive compounds that are responsible for anti-inflammatory, antiasthmatic, hypoglycaemic, vasodialator and other antimicrobial properties. The present investigation aimed to evaluate chemical profile of $H$. spicatum collected from north west Himalaya. Processed rhizomes were analyzed for the presence of metals, alkaloids, fatty acids and volatiles by Atomic Absorption Spectrophotometer and GC-MS combined with HS-SPME. Comparison of data was done with the help of Student’s t-test and is presented as mean ± standard deviation at significance level $p$ ≤ 0.05. Metal/nutrients generally significantly increased in cultivated plant except B (55.50 to 51.60
ppm) and Cl (150.00 to 141.50 ppm). Except linoleic acid (18 : 2ω6 50.00 to 57.33), oleic acid (18 : 3ω9 10.00 to 14.24) and γ-linolenic acid (18 : 3ω6 1.00 to 1.30), all the tested fatty acids significantly decreased in cultivated *H. spicatum*. Only considerable variations were recorded in chemical constituents after three years of experimental cultivation. Acid value ranged from 1.30 to 1.40 while saponification value 108.00 to 111.80 for wild and cultivated rhizomes respectively. There is considerable qualitative variation in the composition of the chemical contents of both the wild and cultivated samples.

Keywords: *Hedychium spicatum*, phytochemical analysis, wild, cultivated, metals.

J. Indian Chem. Soc.,
Vol. 92, January 2015, pp. 111-114

**24,25-Dihydrolimocinol, a new triterpenoid from fresh fruits of *Melia azadirachta***

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*Matured received online 30 September 2013, accepted 18 July 2014*

**Abstract**: A new triterpenoid, identified as 24,25-dihydrolimocinol, has been isolated from fresh fruits of *Melia azadirachta*. Zafaral, which was earlier reported from its leaves, has also been isolated from fruits, this time along with several known limonoids. Their structures were elucidated on the basis of spectroscopic evidences and chemical transformations.

Keywords: *Melia azadirachta* L. syn. *Azadirachta indica* A. Juss., Meliaceae, fresh fruits, triterpenoids, 24,25-dihydrolimocinol, zafaral.

J. Indian Chem. Soc.,
Vol. 92, January 2015, pp. 115-121

**Phytochemicals, heavy metals and antioxidant studies on *Semecarpus anacardium* Linn. (fruit)**

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*Manuscript received online 24 August 2013, accepted 21 July 2014*

**Abstract**: The main objective of the study is to focus on phytochemicals (physicochemical, HPTLC analysis, proximate analysis), heavy metals estimation and antioxidant studies on fruit of *Semecarpus anacardium* Linn. HPTLC method showed that the conc. of catechol in *Semecarpus anacardium* fruit is 0.082% (w/w). The concentration of Cu, Cr, Mn, Zn, Fe, Ni in plant sample were found to 7.71 ppm, 1.13 ppm, 27.25 ppm, 37.51 ppm, 87.71 ppm and 3.71 ppm, respectively whereas Pb and Cd were below detection limit. The proximate analysis showed the percentages of moisture content, ash content, crude protein, crude fibre, crude fat and carbohydrate of *Semecarpus anacardium* fruit as 10.15, 3.21, 8.41, 12.64, 5.30 and 74.87%, respectively while its calorific value is 332.63 kcal/100 g. Free radical scavenging abilities of the methanolic extracts were found to 56.98% by the DPPH method.

Keywords: *Semecarpus anacardium* fruit, phytochemical, heavy metals, HPTLC, proximate, antioxidant.
Volatile constituents of *Geranium wallichianum* D. Don ex Sweet. from north-western Himalayan region

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Manuscript received online 27 November 2013, revised 18 July 2014, accepted 25 July 2014

Abstract : The volatile constituents of *Geranium wallichianum* D. Don ex Sweet. of the family Geraniaceae collected from north-western Himalayan region, India was analyzed by capillary GC and GC/MS. The major volatile constituents were germacrene D (28.5%), β-caryophyllene (15.9%) and selinene <7-epi-α> (8.5%) along with δ-cadinene, α-cadinol, linalool, β-bourbonene, γ-amorphene, α-humulene, caryophyllene oxide, isophytol, β-(E)-farnesene, (E,E) geranyl linalool, terpineol-4-ol, sabinene and cubenol as minor constituents. This is the first report on the essential oil composition of *G. wallichianum* from India.

Keywords : *Geranium wallichianum*, Geraniaceae, essential oil composition, germacrene D, β-caryophyllene, selinene.

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Disposable uric acid biosensor by bacterial crude uricase enzyme modified screen printed electrode

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Manuscript received online 27 January 2014, accepted 31 July 2014

Abstract : Uric acid is the primary end product from purine derivatives in human metabolism. Excessive production of uric acid may lead to gout, hyperuricemia and kidney disorder. Different analytical methods for uric acid such as colorimetry, commercial enzyme electrode and commercially available uric acid kit are used widely. The main purpose of this research was to develop a screen printed electrode based uric acid biosensor using gelatin immobilized uricase enzyme extracted from *Comamonas* sp. BTUA. The enzyme catalyzed oxidation of uric acid in presence of oxygen, producing allantoin and hydrogen peroxide. The linearity of the standard curve in the concentration ranges from 5.94 × 10^{-6} to 4.75 × 10^{-4} molar was satisfactory and could be used for the quantitative determination of uric acid in human serum samples. The limit of detection (LOD) was 2.26 µM and sensitivity was evaluated as 3.31 nA µM^{-1} of uric acid. One modified electrode could be used for six measurements with 95% accuracy up to 25 days. The developed biosensor was easy to use, inexpensive, sensitive and reliable.

Keywords : Uric acid, gout, *Comamonas* sp., uricase, amperometry, screen printed electrode.

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Synthesis and antimicrobial activity of some novel Mannich’s bases of 3-sulfamerazinyl substituted spiro (isatoic anhydrido-4-thiazolidinone) derivatives

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Manuscript received online 27 January 2014, accepted 31 July 2014

Abstract : The aim of this work is to synthesize some novel Mannich’s bases of 3-sulfamerazinyl substituted spiro (isatoic anhydridinone) triazoles. These compounds were characterized by IR, NMR and MS spectra. The biological activity of these compounds was tested against some pathogenic bacteria and fungus strains.

Keywords : Mannich’s base, 3-sulfamerazinyl, spiro (isatoic anhydridinone) triazoles, antimicrobial activity.
Abstract : Synthesis of novel 3'′-[4',N-(4-methyl-2-pyrimidinyl)-benzene sulphonamido]-spiro-(3H-isatoic anhydride-3,2'-thiazolidin)-1H-2,4'-5H)dione (5) and their Mannich's bases 6(a-g) has been described. Treatment of isatoic anhydride (1) with sulphamerazine (2) yielded 4-[1,2-dihydro-2-oxo-3H-isatoic anhydride-3-ylidene]amino]-N-(4-methyl-2-pyrimidinyl)benzene sulfonamide (3). Cyclocondensation of its azomethine function with mercaptoacetic acid (4) over basic alumina afforded 3-[4-N-(4-methyl-2-pyrimidinyl)-benzene sulphonamido]-spiro-(3H-isatoic anhydride-3,2'-thiazolidin)-1H-2,4'-5H)dione (5) which reacted smoothly with secondary amines and formaldehyde to give Mannich’s bases 6(a-g) in excellent yield. Mannich’s bases of sulphamerazinyl substituted [spiro-isatoic anhydride-4-thiazolidinnone] 6(a-g) were screened for their in vitro antimicrobial activities against bacterial species (E. coli and B. subtilis) and fungal species (M. phaseolina and F. oxysporum) by agar-well assay method against the standard drugs (ciprofloxacin for bacteria and fluconazol for fungi).

Keywords : Isatoic anhydride, sulphamerazine, mercaptoacetic acid, Mannich’s bases.


Volatile terpenoid composition and antimicrobial activity of flowers of Melia azedarach Linn. from north west Himalayas, India

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Manuscript received online 29 May 2014, revised 14 June 2014, accepted 07 August 2014

Abstract : Essential oil of Melia azedarach Linn. obtained by hydro distillation using Clevenger apparatus was analyzed by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). Eighteen compounds were identified representing 92.21% of the total oil. The oil contains the highest proportion of oxygenated sesquiterpene hydrocarbons (73.99%). (E)-Nerolidol (70%), n-nonalan (4.78%), (Z)-nerolidol (3.99%) are the predominant compounds along with cumarene (1.34%), n-decanal (1.31%), as minor constituents. In vitro antimicrobial activity of oil was conducted against microbial strains including one Gram-positive (Staphylococcus aureus) and five Gram-negative (Proteus vulgaris, Pseudomonas aeruginosa, Escherichia coli, Salmonella enterica, Klebsiella pneumoniae) bacteria as well as two fungi (Pichia guillermondii, Candida albicans) using agar well diffusion method.

Keywords : Melia azedarach, Meliaceae, essential oil, (E)-nerolidol, antimicrobial activity.


Non-isothermal degradation studies and ion-exchange properties of 4-aminosalicylic acid, urea and formaldehyde copolymer resin

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Abstract: Copolymer resin 4-ASAF was synthesized by the condensation of 4-aminosalicylic acid (4-ASA) and urea (U) with formaldehyde (F) in the presence of 2 M HCl as catalyst with 2 : 1 : 4 molar ratios of reacting monomers. The structure of the resin was characterized by various spectral techniques like infra-red (FTIR) and nuclear magnetic resonance (^1H and ^13C NMR) spectroscopy. The empirical formula and empirical weight of the resin were determined by elemental analysis. The morphological feature of the 4-ASAF copolymer resin was established by Scanning Electron Microscopy (SEM). Thermal study of the resin was carried out to determine its mode of decomposition and relative thermal stability. The Freeman-Carroll, Sharp-Wentworth, Friedman and Change methods have been used in the present investigation to calculate thermal activation energy (E_a), order of reaction (n) and frequency factor (c). The chelating ion-exchange property of this copolymer was studied for eight metal ions viz. Fe^{III}, Cu^{II}, Ni^{II}, Co^{II}, Hg^{II}, Zn^{II}, Cd^{II} and Pb^{II} ions by using batch equilibrium method. The chelating ion-exchange study was carried out over a wide pH range at different time intervals using different electrolyte of various ionic strengths.

Keywords: Copolymer, thermogravimetry, ion-exchange, activation energy.