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CONVERSION OF 2-METHYLCHROMONES TO PYRIMIDINE DERIVATIVES

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(Received March 26, 1995)

2-Methylchromones Ia-d were converted to 4-methyl-6-(substituted phenyl) -2-[1H]-thiopyrimidines IIa-d, via the condensation with thiourea. These thiopyrimidines were converted to other pyrimidine derivatives e.g. bis pyrimidinyl disulphides, oxopyrimidines, ethoxycarbonylmethylmercaptopyrimidines, ethoxycarbonylmethyl-mercaptopyrimidine acid hydrazides, triazolylpyrimidines, pyrimidin-2-ylmercaptoacetyl-thiosemicarbazides and dimethyl oxalacetate 2-carboxymethyl-mercaptopyrimidine acid hydrazides.

Key words: Methyl chromones, Thiopyrimidines, Pyrimidine derivatives.

Introduction

Flavones have been examined to give 2-[1H]-thiopyrimidines [1] on condensation with thiourea. But no 2-methyl-chromones have been treated with the same reagent. When 2-methylchromones I(a-d) were condensed with excess thiourea in alcoholic potassium hydroxide solution 4-Methyl-6-(substituted phenyl)2-[1H]-thiopyrimidines II(a-d) were obtained. All compounds II were soluble in aqueous alkali and gave a colour reaction with ferric chloride; their IR spectra showed bands at 3190-3170 (NH), 2900 (OH. Br), 1625-1620 (C=N) and at 1240-1230 cm^{-1} (C=S). The PMR spectrum of IIb taken as an example showed signals at δ 12.4 (s, 1H, OH), 7.76-6.76 (m, 5H, phenyl and pyrimidine protons), 2.37 (s, 3H, pyrimidine CH_3) and at 2.27 (s, 3H, phenyl CH_3).

Experimental

Melting points were recorded. The IR spectra (potassium bromide) were recorded on a Pye Unicam SP-3100 spectrophotometer. ^1H NMR spectra were measured in DMSO-d_6 on a Varian EM-360 60MHz or Brucker 250 MHz spectrophotometers. Chemical shifts were quoted as δ values in relation to TMS as internal standard.

4-Methyl-6-(substituted phenyl)-2-[1H]-thiopyrimidines II(a-d). A mixture of chromone (0.01 mole), thiourea (0.025 mole), potassium hydroxide (0.02 mole : dissolved in a small amount of water) and ethanol (50 ml) was refluxed for 3 hrs. The solution was cooled and acidified with dilute hydrochloric acid on which a yellow crystalline solid was obtained. It was filtered off and crystallized from the proper solvent to give compounds II.

Bis-[4-methyl-6-(substituted phenyl)-pyrimidin-2-yl] disulphides III (a-d). A mixture of thiopyrimidine II (1g), dioxane (30 ml) and hydrogen peroxide (1 ml, 30%) was heated for 5 mins, then cooled. A white crystalline solid was deposited which was filtered off and recrystallized from the proper solvent to give compounds III.

4-Methyl-6-(substituted phenyl)-2-[1H]-oxopyrimidines IV(a-d). To a solution of thiopyrimidine II (1 g) in 10% aqueous sodium, hydroxide (10 ml), hydrogen peroxide (10 ml, 30%) was added and the mixture was allowed to stand for 24 hrs at room temperature. The yellow sodium salt that separated was filtered off, acidified with dilute hydrochloric acid and recrystallized from the proper solvent to give the oxopyrimidines IV.

2-Ethoxycarbonylmethylmercapto-4-methyl-6-(substituted phenyl) pyrimidines V a and b. A mixture of compound IIa or IIb to (0.01 mole), ethyl chloroacetate (0.012 mole), pyridine (20 ml) and anhydrous sodium carbonate (4 g) was heated under reflux for 10 mins, allowed to cool, then poured into water. A white crystalline solid was obtained which was filtered and crystallized from the proper solvent to give Va or Vb.

2-Carboxymethylmercapto-4-methyl-6-(substituted) pyrimidine hydrazides VIa and b. Compound Va or Vb (0.01 mole), hydrazine hydrate (0.012 mole, 98%) and ethanol (20 ml) were heated on a steam bath for 6 hrs. The colourless crystals which separated on cooling were recrystallized from the proper solvent to give acid hydrazides VIa or b.

2-Carboxymethylmercapto-4-methyl-6-(substituted phenyl) pyrimidine anisylidenehydrazides VIIa and b. A mixture of VIa or VIb (0.001 mole), ethanol (20 ml), acetic acid

Pak. j. sci. ind. res., vol.39, nos.9-12, September-December 1996

MIXED LIGAND COMPLEXES INCLUDING COMPLEXES OF TETRA-AZAMACROCYCLES

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(Received July 18, 1995)

Several new mixed ligand complexes of zirconium (IV), uranium (VI), copper (II) and zinc (II) of compositions, $[Zr(O)Cl_2L^IL^{II}]$ [L^I=o-phenylenediamine, ethylenediamine, 1,6-diaminohexane and L^{II}=quinoline, pyridine pyridine N-oxide and aniline]; $[Zr(O)Cl_2 2L^IL^{II}]$ [L^I=aniline and L^{II}=pyridine N-oxide]; [Zr(O)[14] ane N_4Cl]Cl; $[U(O)_2$ [14] ane N_4] $(NO_3)_2$ and $[ML]X_2$[M=Cu(II) and Zr(II), L=trans-[14] - diene and X=Cl^- and NO_3^-] have been synthesised and characterized. Conductivity measurements reveal that complex 1 is nonelectrolyte and copper (II) and zinc (II) complexes are 2:1 electrolytes in DMSO. But such measurements could not be carried out for other complexes due to their insolubility in almost all common organic solvents. Magnetic measurements, IR and electronic spectral data are consistent with octahedral geometry of zirconium (IV) and uranium (VI) complexes and four-coordinated square planar geometry of copper (II) and zinc (II) complexes.

Key words. Metal complexes, Tetraazamacrocycles, Mixed ligands.

Introduction

Mixed ligand complexes of different metals containing nitrogen and oxygen donors are well-known to serve pharmaceutical [1-3] and industrial interests [4-7]. Transition metal complexes of phthalimide have therapeutic [8,9] and industrial importance [10,12]. Sharma *et al.* [13] have reported a number of mixed ligand complexes of titanium (III) and vanadium (IV) with imides and heterocyclic bases. They have also reported [14] mixed ligand complexes of cobalt (II) and nickel (II) containing diphenic acid as a primary and heterocyclic bases as secondary ligands. Complexes with macrocyclic ligand have received considerable attention, since they exhibit distinctive coordination chemistry and biological activity [14-19]. Hay *et al.* [20,21] have reported a number of cis-complexes of macrocyclic ligands. Considering these facts a study was conducted and mixed ligand complexes of heavier metals, Zr(IV) and U(VI) ions and metal complexes of Cu(II) and, Zn(II) ions containing 14 and 15-membered macrocycles and their syntheses, bonding and structures are reported.

Experimental

Reagents and chemicals. All reagents and chemicals were of reagent grade and used as supplied by E. Merck, except for ethanol which was refluxed with iodine and magnesium turnings, distilled and stored over molecular sieves.

Analyses. Carbon, hydrogen and nitrogen analyses were carried out by the microanalytical services at the University of Erlangen - Nurnberg, Germany.

Preparation of the Complexes: Preparation of (1): $[Zr(O)Cl_2.C_6H_4(NH_2)_2.C_9H_7N]$. $ZrOCl_2.8H_2O$ (0.5 g, 0.0025 mol) was dissolved in absolute ethanol (30 cm^3) to which an ethanolic solution (40 cm^3) of *o*-phenylenediamine (0.27g, 0.0025 mol) and quinoline (0.33 g, 0.0025 mol) was added. The precipitate was filtered, washed with absolute ethanol and dried *in vacuo* over P_4O_{10} to get coffee coloured solid (yield 60%).

Preparation of (2): $[Zr(O)Cl_2.2C_6H_5(NH_2).C_5H_5NO]$. $ZrOCl_2.8H_2O$ (0.5g, 0.0025 mol) was dissolved in absolute ethanol (30 cm^3) to which an ethanolic solution (40 cm^3) of aniline (0.46 g, 0.050 mol) and pyridine N-oxide (0.24g, 0.0025 mol) was added. The blue solid was filtered, washed with absolute ethanol and dried *in vacuo* over P_4O_{10} (yield 65%).

Preparation of (3): $[Zr(O)Cl_2.(en).C_6H_5NH_2]$. $ZrOCl_2.8H_2O$ (0.5 g, 0.0025 mol) was dissolved in absolute ethanol (30 cm^3) to which an ethanolic solution of ethylenediamine (0.15g, 0.0025 mol) and aniline (0.23 g, 0.0025 mol) was added. The precipitate was isolated, washed with absolute ethanol and dried *in vacuo* over P_4O_{10} to get yellowish solid (yield 70%).

Preparation of (4): $[Zr(O)Cl_2.NH_2(CH_2)_6NH_2.C_6H_5NH_2]$. $ZrOCl_2.8H_2O$(0.5g, 0.0025 mol) was dissolved in absolute ethanol (30 cm^3) to which an ethanolic solution (40 cm^3) of 1,6-diaminohexane (0.29 g, 0.0025 mol) and aniline (0.23 g, 0.0025 mol) was added. The precipitate was filtered, washed with absolute ethanol and dried *in vacuo* over P_4O_{10} to get grey solid (yield 62%).

Preparation of (5): $[Zr(O)Cl_2.NH_2(CH_2)_6NH_2.C_9H_7N]$. $ZrOCl_2.8H_2O$ (0.5g, 0.0025 mol) was dissolved in absolute

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Pak. j. sci. ind. res. vol. 39, nos. 9-12, September-December 1996

ACTIVATION OF FULLER'S EARTH OF D.G. KHAN WITH SULPHURIC ACID

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(Received December 26, 1995)

A huge deposit of fuller's earth essentially montmorillonitic has been identified in D.G. Khan. An investigation on its activation with sulphuric acid was made to make it suitable for cleansing and decolourising edible oils. However, special attention was paid to the comprehensive study of (i) concentration of acid (ii) time of activation and (iii) solid contents of the slurry, for activating the clay to a maximum possible degree so as to make it commercially acceptable.

Key words: Activation, Montmorillonite, Clay, Sulphuric acid.

Introduction

Fuller's earth is an important industrial clay mineral with the ability to absorb oil, grease or colouring matter. It is differentiated from the large volume clay mineral products like kaolin, ball clay, fire clay and refractory clay etc in terms of being commercially rare. [1, 2]

A huge deposit of about 10 million metric tons of montmorillinic fuller's earth has been identified in Dera Ghazi Khan [3, 4]. The activation studies of this clay have been undertaken to ensure optimum utilization of the reserve to make it suitable for bleaching and refining edible oils.

The refining of vegetable oils for human consumption involves the removal of a number of impurities phosphatides, fatty acids and trace metals etc, followed by decolourisation and finally deodorisation.The purification and decolourisation stage of refining process employs activated bleaching earth which is later removed from the oils by filtration.

An acid activated clay has proved an efficient decoloriser due to its large surface area produced by the corrosion of the lattice [5]. The cations of the clay absorb colouring matter and impurities onto the surface thus enhancing the bleaching activity. Hence, it was decided that this particular mon-tmorillinic clay be activated with inorganic acid as it serves three purposes: (i) dissolves impurities such as calcite, (ii) replaces exchangeable divalent calcium ions with monovalent hydrogen ions and (iii) dissolves some aluminium ions contained in the tetrahedral layer and some Fe^{+++}, Fe^{++}, Al^{+++} and Mg^{++} ions from the octahedral layer. In tetrahedral layers, some Si^{++++} ions are replaced by Fe^{+++}, Fe^{++}, Ca^{++} and Mg^{++} ions.

Sulphuric acid has been selected because of some advantages over hydrochloric acid; (i) it causes less corrosion problems with mild steel vessels and (ii) produces less fumes, hazardous to health, at activated temperature. The investigations involve comprehensive studies of parameters: (i) concentration of acid (ii) contact time and (iii) clay acid composition, to confirm the suitablility of finished product and its comparability to those of the imported ones available in the market.

At present, the oil industry of our country is relying upon the import of activated clay to meet the rapidly increasing requirements, therefore, a successful attempt on the activation of D.G. Khan deposits of fuller's earth can pave the way for our country to become self sufficient in the activated clay.

Experimental

The experimentation was confined to washing of the clay, its activation and finally the bleaching behaviour of the activated clay toward soybean oil [4].

Washing. The crude clay from the reserve was washed with water in plastic tubs using gravity separating techniques to eliminate small indispersible fraction, called gangue, not suitable for activation. The gangue carried away undissolved impurities and heavy particles settled down. The upper dispersed layer was decanted off, passed through 80 mesh screen in the settling tanks. It was then allowed to stand still until a supernatant water layer separated out which was poured leaving behind washed mud. The mud was spread on clean ground to dry in the sun for further experimentation. The gangue and other impurities were found to be 6.35%.

Activation. The activation of the washed clay was carried out with sulphuric acid according to Table 1. The contents i.e., weighed quantities of acid, clay and water were refluxed in a round bottom flask fitted with water condenser for a specified duration of time. At the end of the reaction, the hot mixture of acid, clay and water was poured down into a plastic tub containing sufficient cold water to stop the reaction. The activated clay was washed with water till the

Pak. j. sci. ind. res., vol. 39, nos. 9-12, September-December 1996

LIPID FRACTIONS AND FATTY ACID COMPOSITION OF *TRIANTHEMA PORTULACASTRUM* LINN

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(Received June 11, 1996)

Trianthema portulacastrum seed oil (12.5 %) has been examined for its physico-chemical characteristics and fatty acid composition. Thin layer chromatography of the oil into lipid classes resulted into polar lipids (4.8%) and neutral lipids (95.2 %). Fractionation of the neutral lipids provided hydrocarbons (0.3 %), wax esters (0.5 %), sterol esters (4.0 %), triglycerides (84.5%), free fatty acids (1.8 %), diglycerides (2.5 %) and monoglycerides (1.6 %). The fatty acids which ranged from C_8 to C_{20} have also been reported.

Key words: Fatty acids, *Trianthema portulacastrum*, lipids, seed oil.

Pak. j. sci. ind. res., vol. 39, nos. 9-12, September-Deember 1996

CHEMICAL COMPOSITION OF THE *CUPRESSUS SEMPERVIRENS* L. FRUITS AT DIFFERENT STAGES OF MATURITY

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(Received October 17, 1996)

The essential oil of *Cupressus sempervirens* L. obtained by hydro-distillation of fruits at two stages was analysed by GC & GC-MS. The main components of the fruits at two stages range from α-pinene (44.59 - 54.62%), sabinene (7.86-2.86%), D^3-carene (15.45-21.83%). a-terpinene (5.25-3.90%). 4-terpineol (6.58-0.04%) and cedrol (1.02-1.35%) respectively.

Key words: Cupressus sempervirens L., Cupressaceae, Monoterpenes, Sesquiterpenes, GC/MS.

Short Communication

Pak. j. sci. ind. res., vol. 39, nos. 9-12, September-December 1996

Complexes of Zr(IV) and U(VI) with Trans- [14]-diene

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Biological Sciences Section

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ELIMINATION OF NATURALLY OCCURRING AFLATOXINS IN COTTONSEED MEAL

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(Received December 21, 1994)

Ammoniation appears to be one of the most extensively studied and applied chemical methods. The present study was aimed at determining optimum time period required at pilot plant scale to detoxify aflatoxins in cottonseed meal by ammoniation under steam pressure and elevated temperature. The detoxification was achieved upto 97% with one percent ammonia at 99 ± 1°C under 0.7± 0.035 bar steam pressure. The quality and texture of starting material was not significantly altered. This study was also aimed to develop a feasible process for detoxification of aflatoxins in cottonseed meal on commercial scale in Pakistan.

Key words: Aflatoxins, Detoxification, Ammonia, Cottonseed meal.

Pak. j. sci. ind. res., vol.39, nos. 9-12, September-December 1996

LIPID STUDIES OF *ANNONA SQUAMOSA*

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(Received 12 June 1995)

The air dried seeds of *Annona squamosa* contain moisture 10.0%, oil 23.0% minerals 1.89% and proteins 24.05%. The fatty acid composition of light yellow coloured oil is C16:0 (13.51%), C18.0 (10.40%), C18.1 (46.96%), C18:2 (25.89%), C18:3 (1.39%) and an unknown acid 1.95% . The unsaponifiable is determined as sterols (25.1%), alcohols (54.1%) and hydrocarbons (20.8%). The hydrocarbons (C11- C31) and alcohols (C16-C18) have been separated, identified ad characterized by the application of thin layer and gas liquid chromatography.

Key words: Annona squamosa, Fatty acids, Hydrocarbons.

Pak. j. sci. ind. res.,vol.39, nos.9-12, September - December 1996

MONITORING THE QUALITY OF SUNFLOWER SEED BEFORE AND DURING STORAGE

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(Received June 12, 1995)

Storage life of sunflower seed can be increased by monitoring the quality of seed before and during the storage period. Cold test, accelerated and modified accelerated aging test, in which several germination tests were made over time were evaluated. Modified accelerated aging test was found to be a more reliable method of seed quality evaluation. Seed lots having P_{50} viability period (period when germination drops to 50%) of 3.5 and above could be stored safely upto one year in conducive environmental conditions.

Key words: Accelerated aging, Viability, Seed vigor, Storage.

Introduction

Pakistan is one of the major edible oil importing countries in the global market. The import of edible oil costs billions of rupees in foreign exchange each year. To reduce the burden of this heavy foreign exchange deficit, many public and private sector seed agencies have taken up production of oilseed crops. Sunflower is one of the major oilseed crops under production in the country as a source of edible oil. Presently, most of the companies are importing hybrid sunflower seeds for crop production.

Sunflower seed, being high in oil content, tends to deteriorate very quickly during storage. It is very difficult to keep sunflower seed in store for a long period of time without substantial deterioration under ordinary storage environments, especially under high moisture and temperature conditions. Viability of seed lots carried over from the previous year is a problem for seed producers. The only solution of this problem at present is to monitor the quality of seed during and after storage.

Several techniques have been developed for the evaluation of seed quality (potential of seed to germinate), but no specific technique is recommended for sunflower seed. In the present study, cold test, accelerated aging test and modified accelerated aging techniques [1-3] are evaluated as indicators of sunflower seed quality.

Materials and Methods

Four hybrids of sunflower seed were obtained from Lever Brothers (Pvt.) Ltd., Lahore and Ghee Corporation of Pakistan. The seed lots were treated with Captan fungicide. Moisture test and standard germination tests were conducted as the seeds were received in laboratory (Table 1).

As no particular vigor test is prescribed for sunflower seed, three vigor tests i.e., cold test, accelerated and modified accelerated aging tests were used for the purpose of evaluation and monitoring the quality of seed.

Cold test. Standard procedures for corn [4] were used for evaluation of sunflower seed. The soil for the cold test was collected from an area where sunflower was grown in the previous season. It was sieved through a 2 mm diameter round-hole screen to remove debris and its moisture content and water holding capacity (i.e., capillary saturation) were determined [4]. Cold tests were carried out in aluminium pans by placing the seeds between 2 cm thick layer of soil. Water cooled to 10°C was then added to bring the medium to 70% saturation. The pans were covered with tight lids and placed at 10°C for 7 days, after which they were transferred to a room maintained at a constant 25°C for 4 days. Cold test emergence was determined by counting the emerged seedlings and calculating the average percent emergence for the four 100 seed replicates.

Accelerated aging test. The test was conducted according to the standard earlier procedure [4]. Seeds were placed one layer deep in the wire mesh tray set on a rack in a plastic box containing 40 ml water (water level was below the wire mesh tray). The box was covered with a tight fitting lid and placed in an incubator at 42°C for 72 hrs. At the end of the aging period, standard germination test (ISTA) was performed

TABLE 1. IDENTITY, INITIAL MOISTURE CONTENT AND GERMINATION PERCENTAGE OF FOUR SEED LOTS OF HYBRID SUNFLOWER USED IN THE STUDIES.

Lot* No.	Source	Initial moisture %	Germination %
1	Lever Brothers (Pvt.) Ltd.	11.5	91A
2	-do-	12.00	92A
3	-do-	11.75	90A
4	Ghee Corporation of Pakistan	11.00	90A

*Lot no. are not actual. These were used only for identification in studies.

Pak. j. sci. ind. res., vol.39, nos.9-12, September-December 1996

EFFECT OF PRE-PLANT INCORPORATED AND POST-EMERGENCE HERBICIDES ON GROWTH AND YIELD OF SUNFLOWER (*Helianthus annuus* L.)

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A field experiment was conducted to study the effect of pre-plant incorporated and post-emergence herbicides on the growth and yield of sunflower cv. Hysun-33, sown in single rows 70 cm apart. Treatments in the experiment were control (weedy check), gramoxone (paraquat) @ 3 lit ha^{-1} as directed post-emergence, Dual 500 E.C. (metolachlor) @ 3 lit ha^{-1} as pre-plant incorporated, Sonalon (ethalfluralin) @ 3.75 lit and 5 lit ha^{-1} pre-plant incorporated. The results revealed that ethalfluralin was the most effective herbicide in controlling weeds and improving sunflower yield of seed oil contents significantly.

Key words: Sunflower, Yield, Herbicides.

Pak. j. sci. ind. res., vol.39, nos. 9-12, September-December 1996

EFFECTS OF SOAKING AND COOKING ON PHYSICAL CHARACTERISTICS, TANNIN CONTENTS AND PROTEIN DIGESTIBILITY OF KIDNEY BEANS (*PHASEOLUS VULGARIS* L.)

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(Received October 5, 1995)

This study reports the effects of soaking and cooking methods on some physical characteristics, tannin contents and in vitro protein digestibility of kidney beans. Soaking of kidney beans in sodium bicarbonate solution gave rise to lower hydration capacity, hydration index, swelling capacity and cooking time than soaking in simple water or neutral sodium chloride solution. Tannin contents of red kidney beans were reduced by soaking at 30° and 100°C for different periods. However, soaking in sodium bicarbonate solution with or without sodium chloride was more efficient in reducing tannin contents. Maximum improvement in protein digestibility was also observed by soaking kidney beans in mild alkaline solutions. Tannin contents were further reduced alongwith improvement in protein digestibility as a result of cooking.

Key words: Kidney beans, Soaking, Cooking, Tannin, In vitro protein digestibility.

Pak. j. sci. ind. res., vol. 39, nos.9-12, September-December 1996

OESOPHAGOSTOMIASIS IN BLACK BENGAL GOATS IN BANGLADESH

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Examination of the intestinal tracts (both small and large) of 208 apparently healthy slaughtered Black Bengal goats from different parts of Bangladesh revealed *Oesophagostomum columbianum* infection in 185 (88.94%) cases. In the presence of the parasites, nodular lesions were detected in the wall of the intestines of 166 (79.81%) goats. The mean worm burden (28.66) and the number of nodules (30.88) varied independently. Both worm burden and nodule formation were found to be very much age dependent ($p<0.01$), higher in goats above 18 months old, but without any seasonal variations ($p>0.05$). Sex of the animals had significant bearing on nodule formation, more in male than in female goats ($p<0.05$). The mucous membrane of the intestines were inflammed, discoloured and covered with mucus. Slightly yellow to green coloured nodules of various sizes were seen on the outer surface of the intestines. There was destruction and desquamation of the villar epithelium due to invasion of parasites. In many cases larval worms were present inside the nodules and destruction of the muscularis mucosa and submucosa was noted. At places, the nodules contained central caseaous mass surrounded by chronic inflammatory cells, mostly lymphocytes and macrophages.

Key words: Oesophagostomiasis, Black Bengal goats, Bangladesh.

Pak. j. sci. ind. res., vol.39, nos.9-12, September-December, 1996

LEAD INDUCED STIMULATION IN THE ACTIVITIES OF PEROXIDASE AND IAA OXIDASE AND THEIR INFLUENCE ON THE GROWTH IN MUNGBEAN

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Foliar application of lead applied in four different concentrations (1, 4, 7 and 10 mM) and the control (0) at five growth stages (28, 35, 42, 49 and 56 DAS) of mungbean resulted in increased activities of both peroxidase and IAA oxidase. Highest stimulation in peroxidase activity was recorded at 56 days after sowing (DAS) by 10 mM dose while IAA oxidase showed maximum activity at 35 DAS under same dose. Lead also caused growth inhibition of plants by decreasing plant height, dry weight of leaf, stem and five entire plants. The effect was pronounced towards higher concentrations of lead.

Key words: Peroxidase, IAA oxidase, Lead nitrate, Mungbean.

Technology Section

Pak. j. sci. ind. res., vol.39, nos. 9-12, September-December 1996

DYES FROM PLANT BIOMASS

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Series of dyes based on sulphur chromophores was manufactured by the baking process involving rice husk from the plant *Oryza sativa* and wood shavings of the plant *Entandrophragma angolense* at 230°C/125 mmHg in a pressure vessel for 3 hrs in presence of aqueous sodium sulphide. Following m-toluene extraction and crystallisation, two dyes were obtained, both of which showed remarkable ultra-violet /visible absorption maxima at λ_{max} of 420nm indicating that they both are of one single colour shade. The dyes gave brilliant yellow colouration of average-good fastness ratings to light and alkaline wash on a bleached and mercerised cotton fabric.

Key words. Dyes, Sulphur chromophores, Plant biomass.