A REINVESTIGATION OF THE CONSTITUENTS OF PEGANUM HARMALA

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(Received October 3, 1963)
BASE CATALYSED HALOGENATION OF ALIPHATIC ALDEHYDES

Part I.—Iodination of Acetaldehyde

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(Received November 29, 1962)

Iodination of acetaldehyde has been carried out in the presence of large excess of alkali. The reaction does not take place strictly according to the equation:

\[ \text{CH}_3\text{CHO} + \text{I}_2 + 4\text{NaOH} = \text{CH}_3\text{I} + \text{HCOONa} + 3\text{NaI} + 3\text{H}_2\text{O} \]

In addition to the normal products predicted by this equation, acetic acid and monoiodoacetic acid were also found, showing thereby that the iodination is followed by the oxidation of acetaldehyde. The low consumption of iodine per mole of acetaldehyde was probably due to some added condensation in the presence of alkali. The rate of iodination was found to be controlled by the order of mixing the reactants and alkali concentration but independent of the iodine concentration.

The haloform reaction has been extensively studied and a large literature, too bulky to be discussed here, has accumulated. Lieben's work on iodination of alcohols and ketones led him to believe that the "haloform test" is given by the compounds containing the CH₂CO-group linked either to carbon or hydrogen. His findings have proved very useful for the detection of water soluble alcohols and methyl ketones. In 1880, Kramer² attempted this reaction for the quantitative estimation of the principal product i.e. haloform by using a gravimetric procedure which is still occasionally used.

Messinger⁴ adopted a volumetric method to study the kinetics of the reaction, which was later on, improved by Hatcher and Mueller⁵ in order to obtain maximum accuracy. However, they found that the reaction never gave the theoretical yield of haloform.

Cullis and Hashmi⁶,⁷ made a detailed study of the base-catalysed iodination of methyl ketones and related compounds. They observed that the reaction does not occur quantitatively according to the equation:

\[ \text{CH}_3\text{COR} + 3\text{I}_2 + 4\text{NaOH} = \text{CH}_3\text{I} + \text{RCOONa} + 3\text{NaI} + 3\text{H}_2\text{O} \]

The present paper deals with the iodination of acetaldehyde which has hitherto remained unexplored. It is found that the overall reaction does not proceed strictly according to the equation (i) under the conditions used by Messinger.⁴

Experimental

Materials.—Commercial sample of acetaldehyde was freshly distilled in a current of nitrogen. The middle fraction was collected and stored in a black-lacquered bottle to prevent photo-decomposition. Acetaldehyde solution was prepared by weight according to Goedwin's procedure⁸ and diluted to the desired strength. The concentration was confirmed by bisulphite method.⁹ All other reagents were "AnalaR" materials.

Kinetic Measurements.—Since this reaction is fairly rapid at 0°C., the rate measurements were carried out in Y-tubes as described by Cullis and Hashmi.⁶ Acetaldehyde and sodium hydroxide were placed in one limb and iodine solution was run into the other. The tubes were closed with rubber bungs covered with tin-foil and then suspended in the thermostat to attain the requisite temperature. The reaction was started by quickly tilting the Y-tube to mix the reagents in a short time. The velocity constants were calculated from the initial slopes of \( \log \left( \frac{V_t - V_a}{V_a} \right) \) vs time curves, where \( V_t \) and \( V_a \) are the volumes of thiosulphate consumed at times \( t \) and infinity, respectively.

Measurement of Total I₂ Consumption.—During this investigation, it has been observed that the order of mixing the reactants does affect the overall consumption of iodine. If the reactants are mixed in the order acetaldehyde—sodium hydroxide iodine, the uptake of iodine was found to be maximum and constant (Fig. t).

Acetaldehyde (25 ml) and varying volumes of sodium hydroxide solution were placed in a well cleaned stopped conical flask and total volume made to 75 ml with water. Iodine solution (25 ml) was added with constant stirring. After a measured period the mixture was acidified with a calculated amount of sulphuric acid, and the liberated iodine was titrated against thiosulphate.
STUDIES IN THE PREPARATION OF OXIDATION-RESISTANT MODIFIED ROSINS

Part I.—Use of Sulphur for the Modification of Rosin and its Esters

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(Received September 10, 1963)

A study of the rosin-sulphur reaction with a view to reducing the oxidation properties of rosin has been carried out. It has been found that the treatment of rosin with sulphur (5%) at 240-250°C. for one hour reduces the oxidation properties of rosin and its esters to negligible proportions. Data of the physical properties and oxygen absorption of the sulphur-treated resins and the methods of treatment are presented in this paper.

Introduction

Rosin is composed of 90% resin acids and 10% non-acidic materials. The resin acids are a mixture of isomers of alkylated hydroaromatic derivatives of 1-methyl-7-isopropylphenanthrene and the structure of the more stable abietic acid is given below.

Abietic acid

The two double bonds in the abietic acid molecule represent a degree of unsaturation which makes the compounds highly unstable. The accepted shortcomings of rosin itself for coating compositions can be attributed for most part to oxidation at the double bonds. In particular, the discolouration and embrittlement characteristics are clearly bound up with its tendency to oxidise. The glaring disadvantages of instability have long been recognised and numerous attempts, which have been made to overcome this drawback, are discussed below.

Dehydroabietic acid

Polymerisation.—Oxidation is greatly reduced because of the conversion of a large part of rosin to a heterogenous dimer. The molecular weight of rosin is increased by about 70% and the polymerised materials have increased melting point, are commercially produced1 and find wide use in the preparation of improved coating compositions.

Hydrogenation.—The use of nickel2 or copper chromite3 as catalyst reduces the melting point of rosin, and therefore the hydrogenated product can be used only in adhesives and rubber industries. The use of catalysts such as Pd, Rh and Ru, however, at 125-300°C. under a pressure of about 3,000 p.s.i.4 or hydrogenations5 of polymerised rosin and subsequent esterification give products having a melting point in the range of 75-85°C.

Oxidation.—Ozonised air has been suggested for oxidation. It gives a dark coloured product having a higher melting point. The oxidised rosin, though soluble in alcohol, ketones, etc. is insoluble in petroleum solvents and this limits its application in coating compositions.

Conversion to Dehydroabietic Acid.—This acid can be prepared by the oxidation of abietic acid with selenium dioxide, followed by dehydration by boiling with acetic acid. Potassium permanganate and bromine have no effect on it. Heat treatment brings about partial conversion of rosin to the more stable dehydroabietic acid, but the temperature proposed for this treatment tends to substantially decarboxylate the molecule. The use of iodine as catalyst has been proposed to effect this conversion at a lower temperature in order to reduce the decarboxylation, but the simplest and probably the most promising way is the treatment with sulphur and this work is mostly covered by patent literature. 6-9.
ION-EXCHANGE FLAME PHOTOMETERIC DETERMINATION OF SODIUM, POTASSIUM, CALCIUM AND MAGNESIUM IN THE ASH OF TOBACCO AND OTHER PLANTS

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(Received July 31, 1963)

Na, K, Ca and Mg in the presence of anions like phosphate and sulphate are estimated here in plant ash using Unicam Sp 900 flame spectro photometer. The interference of the anions and cations with each other has been overcome by using cation-exchange resin. The results are compared with those obtained by conventional procedures.

Introduction

The flame photometer is of particular advantage for the analysis of plant ash since it allows multiple determinations of sodium, potassium, calcium and magnesium in the presence of phosphate and sulphate ions. The classical methods are time-consuming and difficult for an average analytical worker to perform accurately. The radiant energy emissions of the metal ions are affected by the presence of other ions. The error produced may be positive or negative and the amount of the error is dependent upon the concentration and identity of the metal involved. A number of references are found in literature where attempts have been made to determine these elements in the presence of one another.

The presence of potassium decreases the emission of sodium. The effect becomes relatively less as the sodium concentration increases with respect to potassium. The determination of calcium is subject to interference by the presence of magnesium and phosphate. Mosher, Bird and Boyle have reported the depression of calcium emission by larger concentration of magnesium. Calcium of course is separated by precipitation with oxalate. West, Poole and Montgomery have obviated the interfering effect of the diverse ions by the radiation buffers. Parks et al. have shown that phosphate had a pronounced inhibiting effect on the flame intensity of potassium but less effect on sodium. Dipple Bricker and Furman state that the emission intensity of calcium varies linearly and inversely with phosphate concentration. Sugawara, Koyama and Kawasaki separated magnesium from calcium and strontium by precipitating the oxalates of calcium and strontium in the presence of urea. Cooley buffers the phosphate effect by adding an excess of phosphate ion so as to give a limiting depression. Takahashi and Yoshida have compensated the depressive effect of the phosphate ions on the calcium emission by using potassium and also sulphuric acid as radiation buffers. Greweill determined calcium and magnesium in plant materials by the resolution of anion interference by using ethylene diamine tetraacetic acid salts. This integrated procedure works well with potassium, calcium and magnesium, but, in the presence of sodium, calcium has to be removed by precipitation because it causes a positive error. Attempts were also made to remove the phosphate interference by the addition of zirconium nitrate; however this also reduces the calcium emission, as do the phosphates.

In the present work, the interference due to anions like phosphate and sulphate was removed by passing the solution of the ash after the removal of silica through a column of cation exchange resin in the hydrogen form. The phosphate and sulphate ions pass through, while the cations are retained by the column. The interference of the cation with one another has been overcome by elution of the cations from the exchange column selectively with varying strengths of hydrochloric acid. The effect of calcium and magnesium on each other and the effect of sodium on calcium has in this way been completely eliminated, thus making the estimations easier and more accurate than the conventional procedure adopted for flame photometric estimations.

Experimental

In order to establish the conditions for the elution of these cations, known solutions, of Na, K, Ca and Mg were first passed individually through the ion-exchange column and different strengths of HCl were used to elute these cations separately. The eluates were collected in fractions of 10 ml each and the eluates tested for the cation being eluted. In this manner the strength and volume of HCl required to elute a particular ion was established for the length of the column mentioned. However, if the change in the length of column is necessitated by the higher concentration of a particular ion, new elution condition can be easily established. A solution containing a mixture of Na, K, Ca and Mg was then passed through the column. Sodium and calcium were eluted...
STUDIES ON SODIUM HYPOPHOSPHATES

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(Received June 25, 1965)

The oxidation of red phosphorus into sodium hypophosphate with an aqueous extract of bleaching powder was carried out at 12-15°C and an increase in the yield by 15% was noticed. However, when the oxidation was undertaken with sodium chlorite solution, having a small proportion of sodium hypochlorite, the yield was found to be higher than that achieved by the previous methods. Infrared spectra and magnetic susceptibility were determined to elucidate the structure of Na4P2O6·10H2O.

Introduction

Red phosphorus can be oxidised to hypophosphate with different catalysts such as copper nitrate solution, aqueous extract of bleaching powder, sodium chlorite and hypochlorite solutions. None of these methods however give satisfactory yield. It has now been found that the use of aqueous extract of bleaching powder at lower temperatures gives 15% increase in the yield of the hypophosphate. Further improvement has been observed by using a mixture of sodium chlorite and hypochlorite. In the case of the latter the diosodium salt Na2H2P2O7·6H2O was converted into the tetra-sodium salt Na4P2O6·10H2O. The structure of the hypophosphates has been elucidated by studying the infrared spectra and the magnetic susceptibility values of the anhydrous salts.

Experimental

12 g. of red phosphorus were taken in a round-bottomed flask which 120 ml. each of water and 3M acetic acid were added at 12-15°C. The aqueous extract of bleaching powder (prepared by dissolving 150 g. of bleaching powder in 500 ml. of water at 10°C containing 101.53 g/l. of Ca (OCl)2, was poured slowly in the flask. The reaction was complete in about 75 minutes, which could be tested with the litmus paper (if it was bleached in a few seconds, it showed that the reaction was not yet completed). To the colourless filtrate from this, a 15% solution of sodium hydroxide was slowly added (38-50 ml.) until a persistent turbidity appeared. 45 g. of hydrated sodium acetate were added to get a white precipitate of calcium hypophosphate, Ca2P2O7·2H2O. This was filtered and the precipitate was introduced in a mixture of 80 ml. of each of 3N sulphuric acid and ethyl alcohol. The whole mixture was shaken for a few minutes when CaSO4·2H2O was precipitated. It was filtered and hypophosphoric acid was obtained as filtrate. The precipitate was washed with 15 ml. each of 2N sulphuric acid and ethyl alcohol.

45 g. of hydrated sodium acetate were dissolved in the filtrate and a crystalline product, Na2H2P2O7·2H2O, was precipitated along with a little of sodium sulphate. All the orthophosphate remained in the solution as Na2H2P2O7. The product, Na2H2P2O7·2H2O, was filtered off and all the orthophosphate was retained in the filtrate. The compound, Na2H2P2O7·2H2O, was dissolved in 120 ml. of hot water at 70-80°C. and the solution cooled to 40°C. To the solution 15 g. of sodium hydroxide was added. The solution was then allowed to cool gradually which resulted in the precipitation of sodium hypophosphate, Na4P2O6·10H2O. This was filtered and washed first with cold dilute sodium hydroxide and finally with ethyl alcohol. The yield was 16.52 g.

In another attempt sodium chlorite (47.23%) containing a small proportion of sodium hypochlorite (9.13%) was used as an oxidant for the preparation of sodium hypophosphate. The apparatus used in this method was of the under mentioned design.

An outer glass tube called the ‘reaction column’ was 1” in diameter and 12” in length, and another glass tube 25” long and 1 cm. in diameter was inserted in to it. Cold water was circulated through this inner tube to control the reaction. There was a small side-arm opening towards the lower end of the outer tube which was connected to the flask receiving the reaction mixture. Suction may be applied to the flask to draw the solution conveniently. Porcelain chips were placed at the bottom of the reaction chamber followed by an asbestos layer. Alternate layers of red phosphorus and glass beads were then placed over it.

30 g. of red phosphorus was placed in alternate layers with glass beads in the reaction column 500 ml. of sodium chloride solution containing (236.15 g. NaClO3) and 45.65 g. NaClO was taken in dropping funnel clamped above the reaction column. The sodium chloride solution was poured slowly at the rate of 30-48 drops per minute. In order to maintain the temperature in the reaction chamber at 18-23°C. cold water was passed through the central tube throughout the course of the reaction. The flask collecting
RAW LEADLESS OPAQUE GLAZES AT CONE 4

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(Received June 10, 1963)

The investigation covers three aspects of raw leadless, boron-free, opaque glazes maturing at Cone 4. Studies have been made of the effect of (1) the RO fluxes (calcium, barium, magnesium, strontium and zinc oxides) upon fusibility, gloss and texture; (2) the variation of alumina-silica ratio for a promising glaze composition and (3) tin oxide, titania and zirconia, on the opacity and colour. Some typical glaze compositions for bright and mat glazes have been suggested to be tried on an industrial scale. They are the whitest with excellent gloss and opacity.

Introduction

The advantages of lead borosilicate glazes are their easy maturing and high gloss but the presence of poisonous lead and characteristic surface imperfections, however, are objectionable features of these glazes. Boric oxide, along with other soluble constituents, tends to make the glaze soft and somewhat soluble, even after being incorporated in frits. With a view to removing these defects, attempts have been made to develop raw leadless boron-free glazes from the indigenous raw materials. Raw glazes maturing at cone 04-2 have been reported in literature. These glazes contain colamonite as their main flux, but this mineral is not available locally, and it was therefore necessary to explore the use of other minerals for this purpose. Some of the difficulties in formulating the new glazes were in respect of their fusibility but they were, however, eliminated to some extent by the incorporation of suitable fluxes, although, some times at the cost of superior gloss and texture. The present work includes the study of the effect of various fluxes, opacifiers, and variation of alumina-silica ratio for some glazes, upon the fluidity, opacity, gloss and texture of the glazes. The glazes developed during the course of this investigation are suitable for wall tiles and pottery. The desired qualities achieved are economy, simplicity of preparation, ease of application and possibility of single firing.

Materials

Many indigenous raw materials were utilized during the course of this work, along with small quantities of imported chemicals, in order to find some better workable combinations. (1) Indigenous potash felspar with empirical formula

\[ 0.37 \text{K}_2\text{O} + 1.43 \text{Al}_2\text{O}_3 + 7.4 \text{SiO}_2 \]

with a molecular weight of 674 and a fusing temperature of 1200°C. Finely ground native dolomite of molecular formula \(1.0 \text{CaCO}_3 + 0.76 \text{MgCO}_3\) and marble chips were the sources of MgO and CaO. Quartz with 97.5% SiO\(_2\) and Mianwali Clay with 89% clay content were the other materials of this group. (2) Regular grades of some commercial chemicals like BaCO\(_3\), tin oxide, zinc oxide and zircon.

Experimental

1. Plan of Investigation.—After preliminary investigation, a tentatively good glaze, with the following empirical formula was developed:

\[ \begin{align*}
0.24 \text{K}_2\text{O} + 0.45 \text{CaO} \\
0.10 \text{BaO} \\
0.21 \text{ZnO} \\
0.32 \text{Al}_2\text{O}_3 & 3.2 \text{SiO}_2 \\
0.13 \text{ZrO}_2
\end{align*} \]

This composition was chosen as a basis for further investigations. In the first phase of work, the alkali content in the glaze composition was held constant at 0.24 eq. with alumina and silica contents at 0.32 and 3.2 eq. respectively and the effect of the variation in the amount of more than one of the oxides of barium, calcium, magnesium, strontium and zinc were studied. Various glaze compositions thus compounded are shown in Table 1.

In the second phase the effect of the variation of alumina-silica content was studied upon one of the promising glaze composition No. A1, found in the first part of this investigation, as indicated in Table 2.

Finally, glaze No. A11 was again selected in order to study the effect of tin oxide, titanium oxide and zirconia opacifiers, as shown in Table 3.

2. Glaze Preparation.—The individual glazes were prepared by mixing the molecular equivalents of the components of the formulations 2-3. The batches of 1000 g. of dry powdered materials were mixed with the required quantity of water and milled in a laboratory pebble mill for 6 hours to such a fineness that not more than 1% is retained on a 200 mesh screen.
FATTY ACID COMPOSITION OF VARIOUS PARTS OF A POLLED ANGUS STEER*  
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(Received August 16, 1963)  

Fatty acid composition of different parts of a Polled Angus Steer of known age, breed and history was determined by gas-liquid chromatographic analysis. Fatty acids obtained after removal of the unsaponifiable matters from a freshly rendered fat were converted into methyl esters by methanol-acid method. Quantitative estimations of the fatty acids were done from a study of relative retention time. Standards were used separately. Diethylene glycol adipate was used as a stationary phase in a 6 ft. column packed with graded celite. Details concerning procedure used and the results obtained are presented in this paper.

Introduction

Analysis of fatty acids by gas liquid chromatography was first introduced by James and Martin in 1952. Since then several developments have been made and the application of gas liquid chromatographic method of fatty acid analysis has become a matter of routine work. The rate of progress of fatty acid analysis by GLC is so rapid and numerous that it has become very difficult to keep abreast of all the works in this field. The method has proved to be more effective than the best of the conventional methods for estimation and identification of the different components in a mixture of fatty acids. Trace components are now identified quickly and confidently.

The distribution of fatty acids in the body of an animal is not uniform. It has been noted by several workers that the outer fat of pig, sheep and cow contains a higher proportion of unsaturated fatty acids than the inner fat. In a recent study, Ostrander and Dugan have shown in details the distribution of unsaturated fatty acids in the muscular and covering fats of pork, beef, veal and lamb. Even though the variation of fatty acid composition in the different parts of an animal body was observed before no complete analysis of the acids was made. In the present investigation the distribution of both saturated and unsaturated fatty acids from the back, cod, caul, anal and kidney of a known animal is shown.

Experiments and Results

The animal chosen was a two year-old, grass-fed prime Polled Angus steer and was killed at the Southdown Works of the Auckland Farmers' Freezing Company in February, 1961. Since the animal was a steer it was possible to examine the cod fat which is especially formed in the scrotum of the castrated animals. Other samples are kidney fat (hard adipose tissue surrounding kidneys), back fat (skin fat from back), caul fat (attached to intestinal mesenteries) and anal or channel fat (fat surrounding rectum).

The tissues from the different parts of the animal were rendered in a pressure cooker and the fat obtained was washed and dried separately. Melting points were found out by the capillary tube method and iodine values were determined by Wij's method. Each sample has been analysed for the usual constants and the results are given in Table 1.

After saponification of the fat unsaponifiable matters were removed by extraction with diethyl ether. The fatty acids obtained were methylated with a mixture of methanol-sulphuric acid by refluxing on a water bath. The washed and dried methyl esters were analysed by gas liquid chromatography using an eight-foot column packed with graded celite. The solid support was impregnated with diethylene glycol adipate. Argon carrier gas and strontium 90 detector were used. The column was heated to 207°C. for all runs. Chromatograms of the samples are shown in Figs. 1-3.

The fatty acids were estimated by measuring the retention volume of the individual peaks and were expressed in mole percent. Identification of the methyl esters according to the peaks were done from the retention time comparing with known samples which were used separately under identical conditions. Table 2 shows the fatty acid composition of the different samples.

Discussion

Whereas the saponification equivalents and hence the mean molecular weights are essentially the same throughout, vast differences between
THE ROLE OF TRANSPIRATION IN THE ABSORPTION AND TRANSLLOCATION OF MINERAL SALTS IN PLANTS

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(Received July 1, 1964)

Cotton and bean plants were grown in solution culture and during the light period one set was kept in a mineral nutrient solution and another set in distilled water. During the dark period the treatment of two sets was reversed. The concentration of culture solution was such in which the plants grow slightly less well than plants growing in full concentration. This presumably prevented the accumulation of minerals in the root. The height and dry weights of the plants of both the sets were determined at the end of the experiments. The observation indicated that higher transpiration rate during the period of mineral salt availability is responsible for greater absorption of salts and better growth.

Introduction

The precise role of transpiration in the upward translocation of mineral salts in plants has been a subject of considerable discussion. It is known that inorganic ions absorbed by the roots are translocated upwards predominantly in xylem, although the specific channels in xylem, that is, the tracheids, and, in such species as contain trachea, through these cells as well. It follows from this view that an increase in transpiration rate, to the extent that it accelerates the movement of water upwards through these tracheal elements, should similarly increase the rate of transfer of ion from the roots to the shoots.

By exposing corn and bean plants to different humidities to make them vary in the rate of transpiration, Freeland1 and Wright2 demonstrated that an increase in the absorption of water resulted in an increase in the mineral uptake. Hylmo3 has also shown that the degree of absorption of calcium is directly proportional to the amount of water transported in the transpiration stream. The importance of metabolic activities of the root in salt absorption could be taken as one of primary controlling factors but the subsequent movement of solute up into the shoot might be still with the transpiration stream. The experiments of Broyer and Hoagland4 are very important in this respect. They supplied mineral nutrients to three series of barley plants according to the following plan: (1) plants were supplied mineral nutrients during a 12-hour dark period each 24 hours; (2) a second set of plants were supplied with mineral nutrients during the 12 hours of light each day; (3) a third set of plants was supplied with mineral nutrients continuously. At the end of three weeks the concentration of different salts in the roots and shoots as determined.

They concluded, "On the whole, the amount of ions absorbed were roughly the same whether the nutrients were available during the day or only during the night". The concentration of salts in the plants getting nutrients continuously was roughly double that of plants in the other series. Contrary to the first concept, these results suggest that most of the salts absorbed are translocated into the shoot, regardless of the rate of transpiration.

In view of above mentioned conflicting reports it was considered that further studies of this classic problem should be carried out in the light of modern concept of 'outer space' and 'inner space' present in the root.

Materials and Methods

Cotton plants (Gossypium hirsutum L.) and bean plants (Phaseolus vulgaris L.) were used in all the experiments reported in this investigation. Seeds were separated in uniform size lits. After soaking for one hour in distilled water the seeds were placed in culture dishes on moist vermiculite for germination. The culture dishes were placed in an environment room and connected to a compressed air manifold. Sylvania F. 96, TB/CW cool white fluorescent tubes, supplemented with Ken Rad 60-w tungsten bulbs, were used for illumination. The illumination intensity at the top of the plants was about 1000 F.C. A light period of 12-hours at 80°F, was alternated with a 12-hour dark period at 70°F. The relative humidity of the room ranged between 30 and 40 per cent. Mineral nutrient solution was supplied according to the Hoagland's formulation and concentrations given by Meyer et al.,5 were taken as full concentration for the
Studies on the Fungi Occurring as Laboratory Contaminants

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(Received August 15, 1963)

A routine subculture was continued to isolate the fungi occurring as laboratory contaminants. In all, nineteen species have been identified. Most of the isolated fungi belong to the genus Aspergillus. Two species of Aspergillus, viz., Aspergillus nidulans and Aspergillus flavus, definitely belong to Ascomycetes in view of the presence of perithecia containing asc and ascospores. The two fungi of the genus Aspergillus and Sporina which have been considered as varieties of each are Aspergillus niger var. gliseus and Sporina flavispera var. heterospora owing to the proliferation of hyphae and the presence of two types of spores, respectively.

Introduction

It has been observed frequently that the indigenous materials and various intermediates and preparations therefrom on which work is carried out in the various divisions of these laboratories are attacked by fungi. It was therefore considered desirable to identify these fungi and study their life-history and accordingly, a systematic project has been taken up under the title 'Studies on the Fungi Occurring as Laboratory Contaminants'.

A systematic mycological study involves the collection of fungal species from the various sources of infected materials from the different sections of the Laboratories, followed by isolation, identification and observations on the cultural behaviour of the isolated fungi. The work done particularly on the fungi belonging to Ascomycetes, Basidiomycetes and Fungi imperfecti has been described in this paper. The importance of the microbiology of these groups in the fermentation and other chemical industries is well-known. Hence the knowledge of the fungal flora of these groups is likely to be fruitful utilized in the fermentation, food and pharmaceutical industries of East Pakistan. As a matter of fact, one of the isolates from Aspergillus, when studied further for their effect on fermentation, was found to be highly effective.

Experimental

Source of Materials.—Fungi were isolated from laboratory contaminants. They were collected from different types of materials used in these laboratories like sugar solution, plant extract, glycosides and wood chips. Isolates were obtained from a large number of bacterial and other contaminated culture plates.

Methods of Sampling and Isolation.—On getting a report of aerial fungal contaminations in the working materials of other research workers of the Laboratories, the materials were collected and subcultures were made in Czapek's and Waksman's media.

The isolates obtained were purified in two ways. Fungi producing abundant spores were purified by single spore culture. For this purpose the

dilution culture method was followed. A dilute spore suspension was made by shaking vigorously a small mass of spore in a tube of sterile water and then poured on a slide having a thin layer of water agar medium. On placing the slide under microscope, small blocks of agar were cut with the help of sterilized needle. Each block with one spore was transferred to Czapek's or Waksman's medium. Spore producing cultures were purified by pouring very dilute spore suspension in petri-dishes with culture media. After a period of incubation a small number of scattered colonies were seen to appear. Subcultures from these colonies were made.

To purify fungi which did not sporulate readily, hyphal tip culture method was followed by transferring single hyphal tip to culture tubes. For isolations and cultures Czapek's and Waksman's media were used.

Organisms Isolated.—Nineteen species of fungi isolated from different materials as contaminants are dispersed in nine genera representing different groups, namely: Phymocryptos, Ascomycetes and Fungi imperfecti (Table 1)

Among these isolates, Synchphilastrum and Cunninghamella were under Phymocryptos, while the others belong to Fungi imperfecti except for three, viz., two species of Aspergillus and one of Chaetomium.

Penicillium and Aspergillus were the most abundant organisms, the number being 5 and 8, respectively. No Basidiomycetes were isolated.

It is not necessary to give the description of all the species here, as they have already been fully described in the literature. 3–7 However, the six species which showed noticeable variation from the already described species are discussed and discussed here.

Discussion

Aspergillus chevalieri var. proliferus.—Colonies on Czapek's medium restricted, blue green in marginal area with heads and perithecia largely confined to central area. Reverse in yellow to brown. Conidiophores septate up to 49 by 8 μ (Fig. 1). Proliferation of phialides are found in most cases (Fig. 2), one
ANATOMICAL STUDY OF THE STEM AND LEAF OF LIPPIA NODIFLORA MICH

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(Received February 26, 1963)

The macro and microscopic characters of the leaf and stem of Lippia nodiflora Mich, have been described. The young stem is characterized by the presence of collenchyma bands at the ridges while in the older stem the collenchyma encircles the entire cortex. The most distinguishing character of the stem is the presence of unicellular two-armed trichomes on it. The leaf is orbicular but the palisade cells towards the upper epidermis are longer than those towards the lower epidermis. The characteristic feature of the leaf is the presence of large water-storage cells beneath the epidermis and in between the palisade cells. Trichomes resembling the stem trichomes are also present.

Introduction

Lippia nodiflora Mich (Vern-Bukan) belongs to the family Verbenaceae. It occurs in all the tropical and warm temperate regions of the world. It is also widely distributed throughout Indo-Pakistan sub-continent and Ceylon. The whole plant is valued as a diuretic. Infusions of leaves and tender stalks are given to children and to women after delivery. It is also reported to be useful in the treatment of diseases of heart, asthma and bronchitis. It is made into a poultice and used as a maturant for boils. It is also used as a demulcent and as a cooling drug. Two crystalline glycosides and a number of sugars have been isolated from the plant.

As no detailed anatomical studies have been carried out on this plant so far, the present work was undertaken.

Botanical Description

Lippia nodiflora is a small, prostrate, perennial, much-branched herb, extending from 6" to 30", often rooting from the nodes, making large patches. Stem quadrangular, leaves subsessile, cuneate and spathulate, serrate at the broad tip, base attenuate and entire, about one inch in length, opposite and decussate. Flowers pink or white, crowded in axillary, long stalked, oblong head; peduncle is 1-3", with very rarely another from the opposite axil. Bracts 1/10", ovate, shortly acute. Calyx minute, divided into two, hairy. Corolla 1/20" to 1/8"; tube, slender; mouth, 2-lipped, the lower lip being rather longer. Stamens four, in unequal pairs. Fruit dry, minute, separating into 2-1 seeded pyrenes.

Materials and Methods

The material was collected from the experimental farm of North Regional Laboratories, P.C.S.I.R., Peshawar. The identity of the material was checked by comparing with the authentic herbarium specimens available in these Labora-

tories. Pieces of stems and leaves were fixed in F.A.A. for microtome sectioning. The material was then dehydrated by normal butyl alcohol and ethyl alcohol, and the paraffin embedding was done according to Zirkle’s method as given by Youngken. The microtome sections were stained with safranin and fast green. Jeffrey’s method was employed for the maceration of the tissues. Fresh hand sections were cut for the various microscopic tests as given by Johansen and E. Gurr. The uniform powdered material was obtained by sifting it through a No. 60 sieve and studied after clearing in 50% chloral hydrate. Cell measurements were taken with the help of an eye piece micrometer.

Description of the Stem

Macroscopic Characters.—The stem is green and quadrangular and its diameter varies from a few m.m. to 5 m.m. A transversely cut surface of the stem presents a greenish appearance. The amount of xylem as indicated by its creamy appearance comprises about two thirds of the entire diameter of the stem. The odour is slight and the taste is bitter and acrid. (Fig. 1).

Microscopic Characters.—The stem is quadrangular in outline and a large number of two-armed trichomes are present on the surface. The stem is incircled by a single-layered epidermis, the cells of which are cuticularized and are rectangular in shape. The continuity of the epidermis is broken by the presence of stomata in it. The epidermal cells measure 12-28-45 m in length and 24-32-45 m in breadth. In the young stem, beneath the epidermis occurs a band of collenchymatous cells in the region of the ridges. In the older shoots the collenchyma cells form a continuous ring round the cortex (Fig. 2). In the fresh hand section the cell walls of collenchyma are shining while in a stained slide this layer can be differentiated by its deep stain. Below the collenchyma layer is the cortex, the cells of which are thin-walled. Cortical cells in the transverse section measure about 21-38-46 m, while in the tangential
INTRODUCTION

Rhzya stricta Decaisne, (Vern. Vena, Candera) belonging to the family Apocynaceae is a small shrub widely but sparsely distributed from Yemen to the north western region of Indo-Pakistan sub-continent and Sind. The leaves carry a considerable reputation as a bitter tonic for fevers and general debility. The drug is rich in alkaloids (8−10%), of which four have been isolated in pure and crystalline state. Recently a new alkaloid Rhazine has also been reported. As this plant is of medicinal importance and there is no publication existing on this subject, the present work was undertaken.

BOTANICAL DESCRIPTION

The plant is a small, glabrous, very stout, erect and sparingly branched leafy shrub. Leaves yellowish and leathery when dry, sessile. Flower short, axillary, stoutly branched cymes, shortly and stoutly pedicelled. Bracts subulate, persistent. Calyx short, 5 partite, without glands, lobes acute. Corolla white, upper half inflated, salver-shaped, tube cylindrical, throat constricted, hairy. Stamens above the middle of the tube, included, anthers lanceolate, disc annular or obscure. Carpels 2, distinct, style filiform. Follicles erect, parallel, slightly compressed, thinly coriaceous.

MATERIALS AND METHODS

The material used for the present study was obtained from the suburbs of Peshawar i.e. Jamrud and Khyber Agency. The identity of the material was checked in each case by referring to the descriptions available in different floras and was rechecked by comparing them with authentic herbarium specimens available in these Laboratories. Stems and leaves in different stages of development were obtained from plants of different sizes and were fixed in FAA for microtome sectioning. Pieces of stem were softened before dehydration and embedding was done by Lendrum's technique which consists in immersing the tissues in 4% aqueous phenol for one to three days, after washing out the fixative. Dehydration was done with normal butyl alcohol and ethyl alcohol, and paraflin wax embedding was done according to Zirkle's method given by Youngken. The sections were stained with Safranin, and Fast green. For maceration of fresh material Jeffrey's method was employed because this gave very satisfactory results. Hand sections of fresh material were cut for the various micro-chemical tests as given by Johansen and E. Gurr. Quantitative data concerning palisade ratio and stomatal index were obtained by procedures as given by Youngken and analysed statistically. The uniform powdered material was obtained by sifting it through a No. 80 sieve and studied after clearing in chloral hydrate. Cell measurements were taken with an eyepiece micrometer.

DESCRIPTION OF THE STEM

Macroscopic Characters.—The young stem is green and smooth while the older stem is yellowish brown in colour and rough. Leaves are crowded and spirally arranged (Fig. 1). Large number of lenticels are seen on the larger stems. The diameter of the stem varies from about a few mm. to 3-4 cms. Internally the stem presents a creamy appearance. The amount of the xylem as indicated by its yellow colouration comprises about one half of the entire diameter of the stem in young plant. It however, increases as the plant ages and constitutes about three-fourths in 2 to 3-year old stem. The odour is slight, taste bitter and acrid.

Microscopic Characters.—Young stem shows a somewhat circular outline surrounded by a single layered epidermis, the cells of which are cuticulariz-
A CONTRIBUTION TO THE ANATOMY OF TINOSPORA CORDIFOLIA (WILLD) MIERS

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(Received July 4, 1963)

The macroscopic and microscopic characters of the stem and leaf of *Tinospora cordifolia* (Willd) Miers, are described.

The stem is characterised by the presence of a brownish, scaly periderm. There is also a complete ring of sclerenchymatous pericycle encircling the vascular region. Secretory sacs and idioblasts are scattered in the cortex and pith. The vascular bundles retain their separate entity even after secondary growth. The medullary rays are very broad. Vessels are small in length but large in diameter.

The petiole structure differs in different regions. In the basal part it resembles the young stem in having a ring of sclerenchymatous pericycle, while in the upper distal part the pericycle is altogether absent. Leaf shows a typical bifacial structure with a small amount of collenchyma in the midrib. The vascular bundle is enclosed in a bundle sheath. Idioblasts and secretory sacs like stem are also present in the spongy parenchyma.

Introduction

*Tinospora cordifolia* (Willd) Miers belongs to the family Menispermaceae. It is a climbing dioecious vine, distributed throughout the tropical parts of Indo-Pakistan subcontinent. It is a often found cultivated. Stem is bitter, stomachic, antiperiodic, antipyretic and when made into an infusion, used as alterative and aphrodisiac. Starch from stem and root is used as a nutrient, in chronic diarrhoea and chronic dysentery. The juice of fresh plant has diuretic properties and is useful in gonorrhoea. Berberine, crude giloinin and giloinine are the chemical substances reported from it. In the Philippines and Malaya the vine is considered to be a universal medicine. It is commonly prescribed as an aqueous extract in the treatment of stomach trouble, indigestion, diarrhoea, as a tonic in the treatment of ulcer, and as a cure for rheumatism and flatulence in children. It is also used as a febrifuge, vulnerary for itches, internally as an antimalarial agent and externally as a parasiticide. The anatomy of this important medicinal plant has not been worked out previously.

Botanical Description

The plant is a dioecious vine reaching a height of 4-10 meters (Fig. 1). The bark is corky and the shoots are glabrous. The leaves are 5-10 cm. in diameter, alternate, acute or acuminate. The petiole is 4.5-7.5 cm. long. Racemes exceeding the leaves, axillary, terminal or on the old wood.

Material and Methods

The material used in the present study was collected from the cultivated plants growing in the experimental farm of North Regional Laborato-
STUDY OF THE SPECIES OF CLOSTRIDIA INVOLVED IN RETTING OF JUTE

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(Received July 6, 1963)

Several anaerobic bacteria were isolated from retted jute fibres (Corchorus capsularis L.). Of the several isolates, only Clostridium Sp1 and Clostridium Sp2 retted jute stem in the laboratory in relatively much shorter time releasing better fibres.

Introduction

Jute fibre is extracted by steeping jute plants in water to allow certain microorganisms in water to decompose the various gummy and pectic substances cementing the fibres, and thereby release the fibres. This process of extracting jute fibres is known as 'biological retting'. Under natural conditions, biological retting takes about 15-18 days for complete retting.

Various workers have reported different microorganisms involved in the retting of jute. Nandi and Basu reported a few strains of fungi which influence the process of retting. Kayser and Debsarmas have reported a few anaerobic bacteria which can be used for retting these materials. Active aerobic jute retting bacterium (Bacillus polymyxa) has also been reported by Ali.

The present work was undertaken to isolate a few anaerobic bacteria which can ret jute in relatively shorter time, producing quality fibres.

Materials and Methods

The isolations were made from the naturally retted jute fibres collected from various districts of East Pakistan. The cultures were always kept at 37°C. and isolations were made in anaerobic culture jars. After incubation at 37°C. for at least 10 days, the cultures were heat-shocked at 80°C. for 20 minutes to destroy all the nonspore forming vegetative cells so that only the spores of the spore forming survive (spores can withstand heat at 80°C.). They were plated in agar medium (Peptone 5 g., yeast extract 5 g., glucose-D 5 g., L-Cystine 0.2 g. and agar 20 gm./1000 ml. of distilled water), and were placed in anaerobic jar and incubated at 37°C. for 8 days. The different types of colonies obtained were isolated in Peptone-yeast extract broth with agar (0.75 g./litre) and calcium carbonate (only a pinch) which gave better growth and sporulation. After sporulation all the isolates were heat-shocked at 80°C. for 20 minutes. Again they were isolated in broth and liquid paraffin was poured in the broth to attain anaerobiosis.

Sterile jute stem tubes, prepared with slight thioglycolate acid in distilled water, were inoculated with the bacterial cultures under investigation. After inoculation, molten paraffin wax was poured in the tubes to cut off air. The tubes were put inside the anaerobic jars and kept at 37°C. pH of the media was adjusted at 7.2. The media were sterilised for 20 minutes at 15 psi.

Results

The spore forming rod bacteria under anaerobic conditions were species of Clostridia (Clostridium Sp1 and Clostridium Sp2) (Figs. 1-2). These bacteria were the most active types and completely retted the jute stem under laboratory conditions in 5 days. These species were closely related to Clostridium novyi and Clostridium hemolyticum, respectively according to Bergey's manual but with slight variations in their physiological activities. Cultural and Physiological characters of the bacteria are given in Tables 1 and 2.

Discussion

The study of the microorganisms of jute retting tanks in East Pakistan comprised three genera: Bacillus, Micrococcus and Clostridium. In the present investigation four types of isolates were observed. Some of them were complete retters, some were partial retters, some were only bubblers and others were inactive. Only the active and complete retters were chosen for further study. Two species of Clostridia (Clostridium Sp1 and Clostridium Sp2) were isolated and found to be the most active retters of jute. The activity of the isolates were optimum at pH 7.2 and decreased with the decrease of pH value. Whether the retting phenomenon is purely a chemical process or symbiotic
UTILIZATION OF BALCRETE FOAMING AGENT

Part II.—Production of Air-entrained Concrete

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(Received September 12, 1963)

This paper describes the use of 'Balcrete' foaming agent as air-entraining agent for entraining air in cement concrete. The addition of Balcrete, in small quantities, increases the compressive strength of concrete made with less graded aggregates. The increased water-repelling property suggests the use of air-entrained concrete where chemical resistance is required. The other uses of Balcrete as retarder and wetting agent are also briefly discussed.

Introduction

Air-entrained concrete, which has become increasingly popular in the U.S.A. since about 1938, is distinguished from aerated or cellular concrete by the amount of air entrapped in the mixture. The proportion of air in the air-entrained concrete is generally limited to 5 per cent, whereas it is normally much higher in cellular concrete. This type of concrete was discovered as a result of an investigation into the causes of the high frost resistance of the concrete made with certain cements in the north-eastern states of America. The high durability was associated with lower densities and numerous minute bubbles distributed throughout the mass. The cause was traced to the use of resins or fatty materials as grinding aids during the manufacture of Portland cement. The entrainment of air has since been accomplished by adding an air-entraining agent to the mix which results in the dispersion of non-coalescing spheroids of air having diameters from 0.003 to 0.05 inch. The amount of air entrained depends on the quantity of agent added which is either intermixed with the cement during grinding or added as a separate ingredient in the mixer. The latter method is preferred because it permits a greater degree of control. Organic impurities in aggregates and pozzolanas in portland cements influence the amount of air entrained for a given amount of agent. The laboratory work and field trials have proved that this purposeful entrainment increases the resistance of concrete to the disintegrating action of freezing and thawing. The bubbles provide spaces where forces causing deterioration can be dissipated. The entrainment of air reduces the strength of rich mixes more than that of leaner mixes and very lean mixes are slightly increased in strength. The use of 'Balcrete' foaming agent for cellular concrete with neat cement and with cement-sand mixtures has been reported earlier. Its use for the air-entrained concrete and the physical properties of the products are presented in this paper.

Aggregates and Compressive Strength

Entrained air increases workability of the mix and permits the use of less well graded aggregates. Table 1 shows the sieve analysis of Malir river coarse aggregate extensively used in Karachi.
DISCHARGE PHENOMENON AS A TOOL IN THE DYNAMIC MEASUREMENT OF THICKNESS OF THIN OIL FILMS BETWEEN HEAVILY LOADED MACHINE ELEMENTS

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The load-carrying capacity of machine elements, such as gears and roller bearings, depends on the lubricant film thickness that may be sustained between the loaded surfaces. This paper describes one of the methods of direct measurement of oil films called the discharge voltage method and presents a presumptive theory for the discharge phenomenon in oils. Some of the author's results using discharge voltage technique are presented together with his conclusions pertaining to the applicability of the method to running gears. With a better and more comprehensive calibration of discharge voltage, the quantitative interpretation of the results is expected to be reliable.

Introduction

In order to evaluate the load-carrying capacity of gears and other rolling and sliding machine elements, a reliable quantitative method of measurement of lubricant film thickness (of the order of 20 micro-inch) borne by the surfaces is essential. While several methods of reasonable accuracy have been developed for the dynamic measurement of film thicknesses between large discs, 1-3 there is none so far available for direct application to more complicated configurations, such as gear teeth. A promising method is Cameron's voltage discharge method which depends on the discharge phenomenon in oils.

Cameron working with a disc machine collected extensive data to study the effect of applied voltage on oil film resistance. By plotting the resistance against current (Fig. 1) he deduced a simple relationship,

\[ C = R \cdot I \cdot k \]

where \( C \) = constant, designated discharge voltage, \( R \) = resistance, \( I \) = current, \( k \) = constant = 1 for currents above 0.5 amp. Cameron's hypothesis for the mechanism underlying the relationship (A) was that the applied voltage causes a discharge to pass through the oil. The discharge will ensure that any amount of current can pass, as the oil is heavily ionized, such ionization occurring at large currents. At lower values of current, oil behaves as an ohmic resistance as seen from the flatter curves of Fig. 1 with \( k \to 0 \).

Cameron next showed that discharge voltage is sensitive to load (Fig. 2) and to speed, (Fig. 3) and proposed that discharge voltage is in fact a function of the gap between the surfaces, that is, the oil film thickness. Siripongse et al investigating the phenomenon found that when a steady D.C. voltage of about 6V is applied to two metallic surfaces separated by an oil film, discharge voltage-

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Fig. 1.—Oil resistance-current characteristics.

Fig. 2.—Discharge voltage-load characteristics.
SHORT COMMUNICATION

SOME NEW HOSTS OF MACROPHOMINA PHASEOLI (MAUBL.) ASHBY

A. GHAFFAR, A. KARI AND R. MIRZA
Department of Plant Protection, Karachi

(Received May 15, 1963)

EFFECT OF STEAMING ON THE EXTRACTIVES OF GURJAN (DIPTEROCARPUS SPP.)

M. ABDUL LATIF AND W. B. WALLIN
Forest Research Laboratory, Chittagong

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PREPARATION OF COLLOIDAL BISMUTH HYDROXIDE

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Central Laboratories, Pakistan Council of Scientific and Industrial Research, Karachi

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