

Chemical Investigation of Hard Wood Extractives-I.

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SUMMARY

Salai wood (*Boswellia serrate Roxb*) on extraction with ethanol-benzene afforded four components. These components were isolated from the extract by following scheme of Carlberge and Kurth¹ with slight modifications and could be characterized as ellagitannin, oleo-gum-resin, compound identical Liriodenine² and tropolone type compound.

Salai is one of the most useful hard woods used particularly for manufacturing newsprint. No systematic analysis of the extractives of salai wood is reported in the literature. Fowler³, Simonsen³, however have reported the presence of hydrolysable tannins, oleogum resins, volatile oil and polyphenolic compounds in salai without any experimental details. Present work is an attempt to isolate the different constituents from extract by following the scheme of Carlberge and Kurth¹ with slight modifications.

EXPERIMENTAL

Proximate analysis of Salai-wood on O.D. basis was carried out as per TAPPI Standards and the results are tabulated in Table 1.

TABLE—1
PROXIMATE ANALYSIS OF SALAI
(*Boswellia Serrate Roxb*)

S. No.	Determination	Percentage
1.	Ash	4.30
2.	Solubility	
	(i) Cold water	10.40
	(ii) Hot water	6.50
	(iii) Alcohol-Benzene (1:2 v/v)	5.70
	(iv) One per cent Sodium Hydroxide	24.80
3.	Lignin (72% Sulphuric acid)	23.20
4.	Holocellulose (By Ritler and Kurth method)	70.60

EXTRACTION WITH ETHANOL-BENZENE MIXTURE

Salai sample was extracted with ethanol-benzene (1:2 v/v) mixture for 24 hrs with occasional stirring in a Soxhlet apparatus. After removal of solvents, the extract was treated as per Scheme-I for the separation of various components.

ISOLATION AND IDENTIFICATION OF WATER SOLUBLE COMPOUND (A)

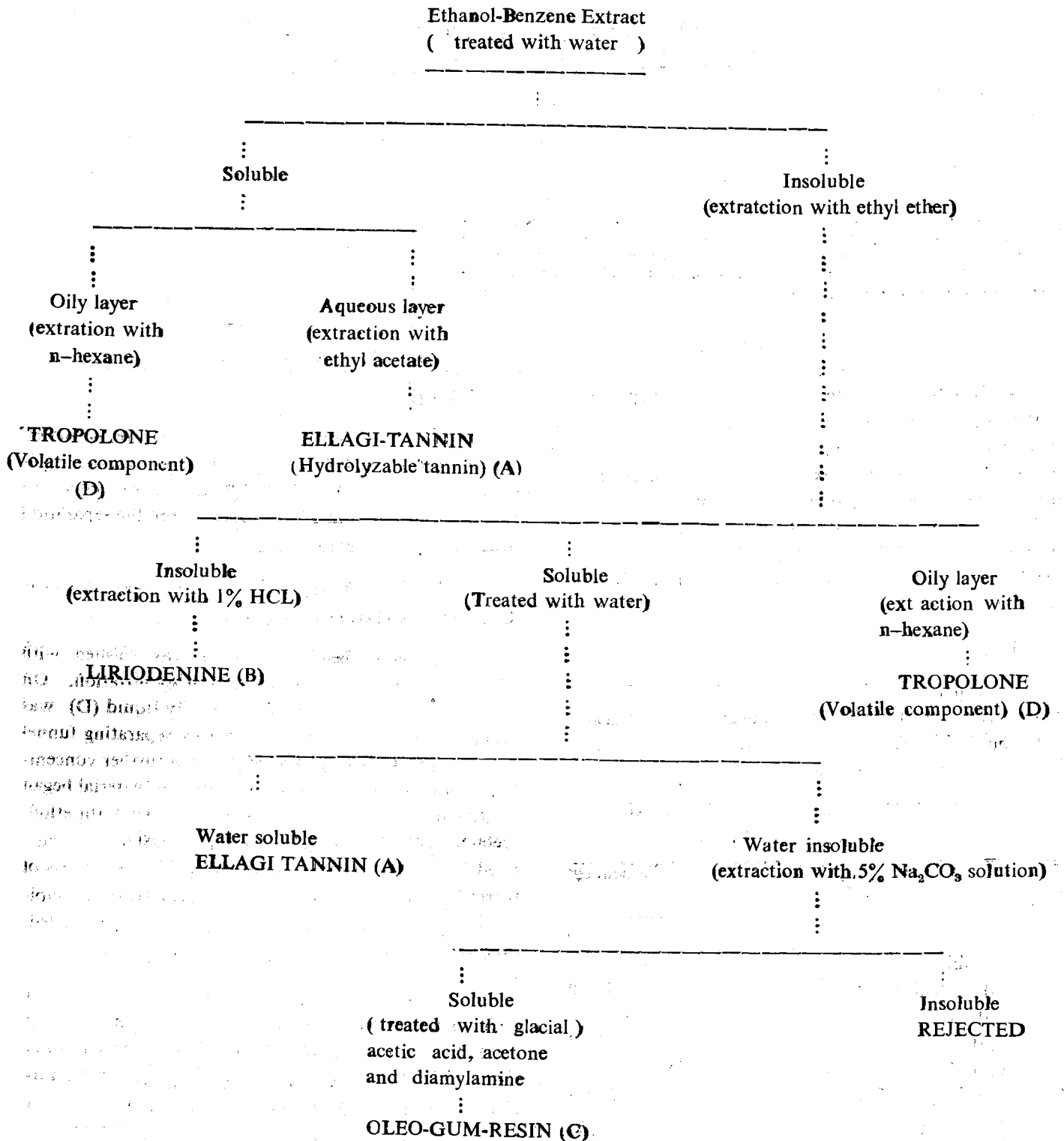
The ethanol benzene extract was washed with water and the aqueous layer removed by filtration. On concentration of aqueous layer, an oily liquid (D) was found to separate. It was separated by separating funnel and kept for further identification. On further concentration of aqueous layer whitish gelatinous material began to separate out which was further extracted with ethyl-acetate. The extract was dried over Na₂SO₄, concentrated to smaller volume and was added to excess of warm hexane when tannin compound (A) was precipitated as an amorphous powder. This was collected, thoroughly with hexane and dried m.p. 235°-237°C.

From the observed properties⁴ of compound (A) such as precipitate with aqueous gelatin and lead acetate; a deep blue colour with ferric chloride and the observed R_f value (0.45) found by paper chromatography^{5,6,7}; it appears that compound (A) is identical

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SCHEME—1

DIAGRAMATIC SCHEME OF CARLBERG AND KURTH'S



with Corilagin⁵ or Juglanin⁷ which is an ellagitannin (hydrolyzable tannin).

ISOLATION AND IDENTIFICATION OF ETHER INSOLUBLE COMPOUND (B)

Compound (B) which was water insoluble part of the original ethanol-benzene extract was isolated by extracting with 1% HCL. The acidic extract was neutralized carefully with solid sodium bicarbonate and the liberated base was extracted with chloroform. The residue obtained on evaporation of chloroform was further crystallised from chloroform when yellow crystalline product m.p. 275°–280°C was obtained.

Qualitative analysis of compound (B) showed the presence of C, H, O and N elements. Compound (B) formed stable rose coloured solution in con. sulfuric acid which changed to yellow on addition of potassium nitrate. When warmed with 1% NaOH containing small amount of sodium hydrosulphite, it formed a blood red solution which changed to yellow on shaking with air; and slowly decolorised to a strong fluorescent solution when boiled with a mixture of aqueous acetic acid, dil hydrochloric acid and tin. All these reactions of compound (B) with reducing agents suggested the presence quinone grouping like that in anthraquinone. The presence of quinone grouping was confirmed by the formation of oxime derivative, m.p. 260 C. These reactions of compound (B) were in agreement with those afforded by the compound liriodenine reported by Buchanan *et al*⁹.

The compound (B) was resolved by two dimensional paper chromatography using butanol-acetic acid-water (60:15:22) v/v followed by 2% acetic acid as solvent system⁸. The well defined spot was then visible under ultraviolet lamp Rf 0.50.

On the basis of quantitative analysis of carbon (74.24%), Hydrogen (3.40%) and Nitrogen (4.98%); empirical formula (CH₁₇gNO₃), was assigned to compound (B)

ISOLATION AND IDENTIFICATION OF ETHER SOLUBLE COMPOUND (C)

The combined ethereal extract collected while isolating compound (B) was worked out to get com-

pound (c) as shown in the scheme. Compound (C) was obtained in the form of amine salt by treating soluble aqueous sodium salt portion successively with glacial acetic acid, acetone and a solution of diamylamine¹⁰, slowly with vigorous agitation. The amine salt of oleo-gum-resin, thus, obtained was crystallized from acetone, m.p. 165°C.

The ethanolic solution of above amine salt was treated with glacial acetic acid and water when free acid was separated, which was crystallised from 95% ethanol, m.p. 170°C. This compound (m.p. 170°C) gave the colour reactions such as Halpher Hicks reaction¹¹, Liberman reaction¹² and reaction with Diazotized p-nitroaniline¹³, indicating it is an oleo-gum-resin acid having abietic diene type skeleton.

Quantitative analysis for elements showed the absence of Nitrogen and Halogens and presence of C, H, O.

On the basis of the results of quantitative analysis for carbon (78.40%), and Hydrogen (10.20%); the empirical formula C₂₀H₃₀O₂ was assigned to compound (C).

ISOLATION AND IDENTIFICATION OF OILY COMPONENT (D)

The oily liquid which was separated during the process of isolation of compound (A) and (B) was shaken with 5 ml of n-hexane.

An oily compound gave intense brilliant and colour with ferric acetate¹⁴, whereas with cupric salt¹⁴, it gave green colour. Attempts to obtain any crystalline material from oily liquid was unsuccessful. However, the volatile nature and the colour reactions are suggestive of Tropolone^{3,14,15} type compound.

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