

QUANTIFICATION OF SOME COMPONENTS OF THE EXTRACTIVES OF *DRYOBALANOPS AROMATICA* OBTAINED FROM DIFFERENT SOURCES

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RASADAH MAT ALI, KOH, M.P. & MICHAEL, M. 1991. Quantification of some components of the extractives of *Dryobalanops aromatica* obtained from different sources. The chemical components of kapur (*Dryobalanops aromatica*) extracts were separated and quantified by a simple and rapid High Performance Liquid Chromatography (HPLC). Eluting with ethylacetate : hexane (3:7) using Li Chrosorb Diol Silica gel column provided a clear separation of dipterocarpol, B-sitosterol, dryobalanone, w-hydroxyfatty acid ferulate, kapurone and terpinylhydrate.

Key words: Quantification - extractives - *Dryobalanops aromatica* - HPLC

Introduction

The genus *Dryobalanops* belongs to the family Dipterocarpaceae. This genus consists of nine species which are distributed widely in Sumatra, Peninsular Malaysia and Borneo. Two species that can be found in Peninsular Malaysia are *Dryobalanops aromatica* and *Dryobalanops oblongifolia* (Corner 1952). *D. aromatica*, known locally as kapur is a valuable timber tree and also a source of camphor (Corner 1952).

Gluing difficulties have often been observed when kapur is solely used for making plywood (Wellon *et al.* 1977, Abe & Ono 1980). One of the reasons for the difficulties may be due to the extractives which retard the curing of adhesives. In order to verify this possibility, a study on the extractive content of kapur was carried out. The occurrence of the dammarane type triterpenes such as terpinylhydrate, kapurone, dipterocarpol and dryobalanone in kapur has been reported (Immamura *et al.* 1970).

This paper describes the determination and quantification of four dammarane type triterpenes, a sterol and an acid obtained from the wood of kapur using HPLC. To date, the quantification of *D. aromatica* has not been reported.

Experimental

Analytical HPLC was carried out with a Hewlett-Packard Model 1084B liquid chromatograph on a Li Chrosorb Diol Silica Gel (10 μ m particle size) stainless-steel column (25 x 0.46 cm i.d.) eluted isocratically with ethylacetate : hexane (3:7). A flow rate of 1.5 ml min⁻¹ and an average pressure of 400 p.s.i at 30°C were employed. The peaks were detected using a refractive index detector.

Calibrating HPLC column

A standard solution containing 21 mg kapurone, 18 mg ragdipterocarpol, 37 mg terpinylhydrate, 8 mg dryobalanone, 91 mg B-sitosterol and 26 mg w-hydroxyfatty acid ferulate was prepared in 2 ml ethylacetate : hexane (3:7). Methanol (0.5 ml) was added to this mixture as an internal standard.

Portions (10 μ l) of the dilute standard solution were used. The ratios of peak areas and retention times of the components were correlated to their respective concentrations.

Preparation of samples

The wood materials used in this study were obtained from three different sources, that is Forest Research Institute of Malaysia (FRIM), PERMINT Plywood Co. Bhd., Kuala Terengganu and Lesong Forest Product, Kuala Rompin, Peninsular Malaysia. Kapur samples from FRIM were obtained from a 54-y-old tree. The samples from PERMINT were obtained in the form of rotary cut veneers. These veneers were classified as face veneers (sapwood) and core veneers (heartwood). The LESONG samples were 5 mm thick discs cut from the butt end of the logs.

The air dried wood samples (10 g) were powdered and extracted successively with hexane (200 ml) and ether (200 ml) in a soxhlet extractor for 8 h. The extract was concentrated to dryness using a rotary evaporator. A mixture of ethylacetate : hexane (3:7) was used to dissolve the extract. Methanol (0.5 ml) was added as an internal standard. The resultant mixture was allowed to stand for 10 min and the supernatant liquid filtered through a 0.45 μ m filter. The filtrate (10 μ l) was injected into the column and developed with ethylacetate : hexane (3:7). Compounds identification was carried out by comparison of the retention times (Rt) with those of the standards and by co-chromatography on TLC plates with added standards. The results of HPLC analysis are shown in Tables 1 and 2.

Results and discussion

The kapur wood extracts contained largely terpenes and fatty acids. Some of the components identified and quantified by HPLC were hydroxydammarones-11, B-sitosterol, dryobalanone, w-hydroxyfatty acid ferulate, kapurone and terpinylhydrate.

From the hexane extract (Table 1), it was found that samples from FRIM, face/back (F/B) and core veneers of PERMINT had similar chemical composition with w-hydroxyfatty acid ferulate being the major component. FRIM samples had, on the whole, a lower percentage of identified components compared with those from PERMINT (F/B and core) and LESONG FOREST PRODUCT. Terpinhydrate (compound 5) could not be detected in the samples from FRIM and PERMINT.

Table 1. Chemical composition of the *Dryobalanops aromatica* samples (%o, w/w) obtained from different sources (Hexane extracts)

Source of samples (Plant part)	Yield of crude extract (%) (w/w)	Components (Retention time, Rt)						
		1 (3.17)	2 (3.24)	3 (3.49)	4 (6.83)	5 (8.63)	6 (8.84)	7 (-)
PERMINT								
a) Core	0.37	23.81	16.12	3.75	4.55	Tr	37.95	31.88
b) F/B	0.20	17.98	13.81	34.31	0.61	Tr	33.21	0.09
FRIM	0.23	0.67	4.86	1.47	4.76	Tr	11.11	77.14
LESONG								
a) Oct 1988								
H/W	0.41	Tr	9.32	11.74	Tr	8.67	53.27	17.00
S/W	0.19	Tr	29.01	51.48	Tr	5.31	Tr	14.20
b) Nov 1988								
H/W	0.58	44.73	5.33	6.54	Tr	Tr	Tr	43.47
S/W	0.41	62.35	9.19	8.16	Tr	Tr	Tr	20.30
c) March 1989								
H/W	0.28	34.41	16.32	4.77	2.71	Tr	35.66	6.13
S/W	0.32	32.39	16.16	Tr	1.31	Tr	35.53	14.16

Abbreviations: H/W: Heartwood; S/W: Sapwood; Core : Veneers for plywood core; F/B : Veneers for plywood faces; Tr : Trace (<0.01%); 1 = Hydroxydammarones-11 (Dipterocarpol); 2 = Kapurone; 3 = B-sitosterol; 4 = Dryobalanone; 5 = Terpinhydrate; 6 = w-hydroxyfatty acid ferulate; 7 = Unresolved fraction; (abbreviations follow for Table 2)

As seen from Table 1, kapurone (compound 2) was detected in all the sample studied. Amongst all the samples collected, the highest percentage of kapurone was detected in the sapwood of LESONG samples collected in October that is, 29%. Among the LESONG samples, the log collected in March had the highest number of identifiable components. This may be attributed to the lower loss of extractives to the environment as compared to the October and November logs. Dipterocarpol (compound 1) was also one of the major components in the March log. There was a marked decrease of dryobalanone (compound 4) as compared to the samples from both PERMINT and FRIM samples.

The ether extraction of the LESONG samples gave an overall much higher yield in crude extracts and also a higher percentage of the identifiable components (Table 2). Most significant is the relatively higher kapurone content and the negligible quantity of w-hydroxyfatty acid ferulate. Terpinhydrate could be detected in the PERMINT (core) samples and in both the November and March logs from LESONG FOREST PRODUCT. As shown

in Table 2, all the six components could only be quantified in the PERMINT (core) samples. Among the LESONG samples, sapwood and heartwood showed no significant variation in component composition.

Table 2. Chemical composition of the *Dryobalanops aromalica* samples (% w/w) obtained from different sources (Ether extract)

Source of sample (Plant part)	Yield of crude extract (%) (w/w)	Component (Retention time, Rt)						
		1 (3.17)	2 (3.24)	3 (3.49)	4 (6.83)	5 (8.63)	6 (8.84)	7 (-)
PERMINT								
a) Core	0.22	0.97	9.11	4.16	0.22	39.39	20.17	25.9
b) F/B	0.46	Tr	0.74	4.59	0.24	Tr	14.21	80.2
FRIM	0.22	4.21	4.43	7.07	0.55	Tr	8.09	75.6
LESONG								
a) Oct 1988								
H/W	0.43	9.59	30.01	Tr	Tr	Tr	Tr	60.4
S/W	0.27	25.16	23.13	27.41	-	Tr	Tr	24.3
b) Nov 1988								
H/W	0.68	21.41	22.66	24.01	1.89	24.68	Tr	5.3
S/W	0.52	3.14	-	7.01	Tr	Tr	Tr	89.0
c) March 1989								
H/W	0.34	7.38	35.68	Tr	0.54	Tr	6.78	49.6
S/W	0.16	5.01	2.21	0.91	Tr	31.49	Tr	60.3

The occurrence of the dammarane type triterpenes such as terpinhydrate, kapurone, dipterocarpol and drybalanone has been published (Imamura *et al.* 1970). In this study, the relative amounts of the extractives vary depending on the age of the tree, the growth environment of the particular tree, the location of the tree as well as the location in the tree where the wood sample is obtained.

Conclusion

From the hexane extract, it was found that samples from the three locations (FRIM, PERMINT and LESONG FOREST PRODUCTS) had about the same major components. The FRIM sample had a lower percentage of the identified components as compared with those from PERMINT and LESONG FOREST PRODUCTS. The ether extract of the LESONG samples gave an overall higher extractives yield and also a higher percentage of the identifiable components.

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