



Chemical investigation of semi purified shorea robusta gum resin by using GCMS

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ABSTRACT

The chemical investigation was done on Semi purified Shorea robusta gum resin (SG) to identify the chemical moieties. N-hexane fraction Shorea robusta gum, fractions were done by using column chromatography. The fractionation done by changing the polarities of n-Hexane and chloroform. All the fractions were collected carefully and solvents were removed by using Buchi Rotavapor. The collected fractions were subjected for GCMS evaluation. This study reveals the probable chemical components which were presenting in each and every fractions based on mass spectrums of GCMS. In the semi fractions of gum resin of Shorea robusta, the 100% hexane fraction was shown good emulsifying property. Based on the GCMS data, the probability of the molecular formulas of the components in 100% hexane fraction were C₉H₁₂, C₁₃H₂₈, C₁₄H₃₀, C₁₀H₂₂, C₁₁H₂₄, C₁₂H₂₆, C₈H₁₈, C₉H₁₉I, C₈H₁₆O, C₆H₁₄, C₁₅H₁₈O₂, C₉H₁₄O₂, C₁₅H₂₄, C₁₆H₃₄, C₁₇H₃₆, C₁₄H₃₀O₃S, C₁₄H₃₁BO, C₁₅H₂₆O. This study gave the information about the probability of molecular formulas and its molecular structures of the components present in the semi fractions of gum resin of Shorea robusta based on GCMS data.

Keywords: GCMS, BPX-70 GC capillary column, Molecular structures, Molecular formulas.

INTRODUCTION

An emulsion is a dispersion of droplets of immiscible liquid in a continuous phase. An emulsifier is a substance that stabilizes an emulsion by different chemical and physical mechanisms, such as surface tension, repulsion and viscosity modification [1][2]. The majority of cosmetic products are emulsions. Based on safety concerns compare with synthetic emulsifiers, natural emulsifiers are most useful amyrenone and b-amyryn. The gum resin of shorea

robusta has lot of components like triterpenoids, ursolic acid, amyrenone, and b-amyryn along with polyphenols and carbohydrates [3]. Some of the studies are available for analysis of gum resin by chromatography [4][5]. The semi purified fraction of *Shorea robusta* with 100% hexane shows good emulsifying property, the remaining fractions with mixer of chloroform and hexane shows poor emulsifying properties. The intense of this study was to find out the probable chemical compounds which

are present in each and every fractions of *Shorea robusta* gum resin. The present study was concerned the analysis of semi fractions of gum resin of *Shorea robusta* by GCMS.

MATERIALS AND APPARATUS

Apparatus

Gas Chromatography with AOC-20i Auto injector (model: GC-2010, make: Shimadzu), Mass (model: GCMS-QP2010 Plus, make: Shimadzu) Column-BPX 70 (Length-30m, Diam-0.25mm, Film-0.25 μ m, Make: SGE), Sonicator (model: Soltec, Make: Sonica), Electronic Weighing Balance (Model: ML204 IA01, Make: Mettler Toledo), Volumetric flasks (100 \pm 0.2mL, Make: Borosil).

Chemicals and Reagents

Chloroform (AR grade) (Make: Rankem), n-Hexane (HPLC grade) (Make: Merck), the gum resin of *shorea robusta* was procured from local market by The Himalaya drug company.

INSTRUMENTATION

GC Conditions

BPX70 capillary column (Length: 30m, Inner diameter: 0.25mm, Film: 0.25 μ m), Carrier gas: Nitrogen, Injection temperature: 250 $^{\circ}$ C, Column flow: 1.62mL/minute, Split ratio: 1:30, GC program: Initial 70 $^{\circ}$ C hold 5minutes, increase 10 $^{\circ}$ C/minute up to 120 $^{\circ}$ C then hold 5 minutes and increase 10 $^{\circ}$ C/minute up to 240 $^{\circ}$ C then hold 5 minutes, injection volume: 5 μ L.

GCMS Conditions

Ion source temperature: 250 $^{\circ}$ C, Interface temp: 200 $^{\circ}$ C, Solvent cut time: 2.0 mm, Detector gain mode: Relative, Detector gain: 0.0 kV, Threshold: 1000, ACQ mode: Scan, Event time: 0.50sec, Scan speed: 1666, Mass range (m/z): 40 to 800.

EXPERIMENTAL

The gum resin of *shorea robusta* was taken and did the purification by column chromatography using the solvents methanol, hexane, chloroform, ethyl acetate and its combinations and collected the fractions.

Dried gum resin of *Shorea robusta* was powdered and purified by column chromatography with solvents. Collected fractions were concentrated under reduced

pressure using a Buchi Rotavapor. The dried fractions were used as emulsifier.

For this semi purification process, we were taken solvents 100% hexane and the combinations 95%chloroform + 5% hexane, 90%chloroform + 10% hexane, 85% chloroform + 15% hexane, 80%chloroform + 20% hexane, 70%chloroform + 30% hexane, 50%chloroform + 50% hexane, 25%chloroform +75% hexane. These fractions were used for GCMS analysis.

1% solution (1g in 100mL) of these fractions in chloroform were prepared and injected for GCMS analysis. Based on these GCMS data, we found the probability of the molecular formulas which were presented in the fractions of gum resin of *Shorea robusta* were C₆H₁₈Se₃Sn₃, C₉H₁₂, C₇H₁₄O, C₁₂H₂₆, C₁₃H₂₈, C₁₁H₂₄, C₈H₁₈, C₆H₁₄, C₈H₁₆O, C₉H₁₉I, C₁₀H₂₀O, C₁₁H₂₃I, C₁₇H₃₆, C₁₄H₃₀O₃S, C₁₅H₃₂, C₁₂H₂₅Br, C₇H₁₂O₂, C₁₄H₃₁BO, C₆H₁₂O₂, C₉H₂₀, C₂H₅NO₂, C₉H₁₈S, C₇H₁₄O, C₆H₁₀O₂, C₇H₁₆O, C₁₅H₂₀O₃, C₇H₁₆, C₁₀H₂₂, C₈H₁₆O₂, C₁₃H₂₇I, C₁₂H₂₅I, C₁₄H₂₉I, C₉H₁₆O₄, C₈H₁₈N₂, C₁₃H₂₂O, C₁₂H₁₉NO₃, C₁₀H₁₈, C₁₄H₃₀O₃S, C₁₆H₄₈O₁₀Si₉, C₁₄H₄₂O₅Si₆, C₁₃H₄₀O₅Si₆, C₁₆H₅₀O₇Si₈, C₁₆H₄₈O₆Si₇, C₇H₁₀O₃, C₁₀H₁₅NO₃, C₁₅H₂₆O, C₁₈H₃₂O₂, C₁₄H₂₂, C₁₅H₁₈O₂, C₉H₁₄O₂, C₁₅H₂₄, C₁₂H₁₈, C₁₃H₂₀, C₁₁H₁₈N₂, C₁₂H₂₀, C₁₇H₁₉NO, C₉H₁₄O₅, C₁₈H₃₀, C₈H₁₂, C₁₁H₁₆O₂, C₁₀H₁₆, C₁₅H₁₉BO₂, C₁₇H₁₆CINO₃, C₁₂H₁₇Br, C₁₄H₂₂, C₁₀H₁₈, C₁₅H₂₆O, C₁₅H₂₄.

In the semi fractions of gum resin of *Shorea robusta*, 100% hexane fraction was shown good emulsifying properties and remaining fractions were shown poor emulsifying properties. The 100% hexane fraction of gum resin of *Shorea robusta* is used as a natural emulsifier in cosmetic products. Based on GCMS data we found the probability of the molecular structures of the components, which were presented in 100% hexane were C₉H₁₂, C₁₃H₂₈, C₁₄H₃₀, C₁₀H₂₂, C₁₁H₂₄, C₁₂H₂₆, C₈H₁₈, C₉H₁₉I, C₈H₁₆O, C₆H₁₄, C₁₅H₁₈O₂, C₉H₁₄O₂, C₁₅H₂₄, C₁₆H₃₄, C₁₇H₃₆, C₁₄H₃₀O₃S, C₁₄H₃₁BO, C₁₅H₂₆O. Based on the GCMS data the components which are presented in the 100% hexane fraction the probability of the names and the components purities are given in table1.

| Retention time | Purity (% of Area) | Probability of the component molecular formula | Probability of the component name |
|----------------|--------------------|--|---|
| 4.9 | 23.49 | C9H12 | 1,2,4-Trimethylbenzene., 1,3,5-Trimethylbenzene |
| 7.2 | 8.34 | C13H28 | 4,7-Dimethylundecane., 3,7-Dimethylundecane., 2,8-Dimethylundecane |
| 10.4 | 4.60 | C13H28, C14H30, C10H22, C11H24 | n-Tridecane., n-Tetradecane., n-Decane., n-Undecane |
| 11.8 | 2.88 | C12H26, C8H18, C13H28 | 3,4,5,6-Tetramethyloctane., 3,3-dimethylhexane., 3,7-Dimethylundecane |
| 12.2 | 7.27 | C12H26, C13H28 | 3,7-Dimethyldecane., 3,7-Dimethylundecane., 2,3,7-Trimethyldecane, |
| 13.6 | 1.89 | C9H19I, C8H16O, C6H14 | 1-n-Nonyl iodide., 3-Methyl-4-heptanone., 2,2-dimethylbutane |
| 14.9 | 1.71 | C15H18O2, C9H14O2 | 2-methylcyclohex-2-enyl ester., Methyl 2-methyl-3-methylenecyclopentanecarboxylate |
| 15.7 | 6.17 | C15H24 | Copaene., alpha-Cubebene |
| 16.1 | 18.56 | C15H24, | Cyclohexane., 2,4-diisopropenyl-1-methyl-1-vinyl, 2,4-Diisopropenyl-1-methyl-1-vinylcyclohexane |
| 16.5 | 6.36 | C16H34, C13H28, C17H36 | n-Hexadecane., n-Tridecane., n-Heptadecane |
| 18.4 | 4.28 | C15H24 | Germacrene D., Copaene., 1,2,3,4,4a,5,6,8a-octahydro-7-methyl-4-methylene-1-(1-methylethyl)-Naphthalene |
| 18.6 | 7.24 | C14H30O3S, C14H30 | Sulphurous acid, 2-ethylhexyl isohexyl ester., 4,6-Dimethyldodecane |
| 18.8 | 2.73 | C14H30O3S, C9H19I, C14H31BO | Sulphurous acid, 2-ethylhexyl ester., n-Nonyl iodide., Diethyl(decyloxy)-borane |
| 19.2 | 1.64 | C15H26O, C15H24 | Dihydro-cis-.alpha.-copaene-8-ol., 1H-Cyclopenta[1,3]cyclopropa[1,2]benzene, octahydro-7-methyl-3-methylene-4-(1-methylethyl)-, [3aS-(3a.alpha., 3b.beta.,4.beta., 7.alpha.,7aS,. Tetracyclo[6.1.0.0(2,4).0(5,7)] nonane,3,3,6,6,9,9-hexamethyl-(1.alpha.,2.alpha.,4.alpha., 5.beta.,7.beta., 8.alpha.) |
| 19.6 | 2.85 | C9H19I, C6H14, C8H17I, C8H16O | n-Nonyl iodide., 2,2-dimethyl-butane., n-Octyl iodide., 3-Methyl-4-heptanone |

Table1: 100% hexane semi fraction of gum resin of *Sohrea robusta* GCMS data

The expected molecular formulas of the components which are presented in the fractions of gum resin of *Shorea robusta* are shown in table 2.

| Sl.No | Fraction of gum resin of <i>Shorea robusta</i> | Expected Molecular formulas of the components |
|-------|--|--|
| 1 | 5%hexane + 95% chloroform | C8H16O, C6H12O2, C9H20, C2H5NO2, C9H18S, C7H14O, C6H10O2, C7H16O, C9H12, C15H20O3, C14H18O3, C13H28, C7H16, C12H26, C11H23I, C8H17I, C6H14, C10H22, C11H24, C10H20O, C8H16O2, C15H32, C13H27I, C12H25I. |
| 2 | 10%hexane + 90% chloroform | C9H12, C14H29I, C6H14, C11H23I, C17H36, C16H34, C13H28, C20H42, C14H30, C15H32, C18H38, C13H27I, C12H25I. |
| 3 | 15%hexane + 85% chloroform | C7H14O, C9H12, C12H26, C13H28, C11H24, C8H18, C12H26, C6H14, C8H16O, C9H19I, C10H20O, C11H23I, C17H36, C14H30O3S, C15H32, C12H25Br, C7H12O2, C14H31BO. |
| 4 | 20%hexane +80% chloroform | C7H14O, C6H12O, C9H12, C13H28, C10H22, C9H19I, C8H18, C6H14, C12H26, C8H16O, C7H16, C9H16O4, C8H18N2, C8H16O2, C7H12O2, C9H20, C13H22O, C12H19NO3, C10H18, C10H20O, C14H31BO, C14H30O3S, C16H48O10Si9, C14H42O5Si6, C13H40O5Si6, C16H48O6Si7, C16H48O8Si8. |
| 5 | 30%hexane + 70% chloroform | C8H16O, C6H12O, C7H10O3, C7H14O, C9H12, C13H28, C11H24, C12H26, C17H36, C14H30O3S, C14H30, C10H22, C19H36, C13H24O, C10H15NO3, C15H32, C15H26O, C10H18, C18H32O2, C15H24, C18H54O9Si9, C16H48O6Si7. |
| 6 | 50%hexane +50% chloroform | C9H12, C13H28, C12H26, C6H14, C8H16O, C7H12O2, C10H22, C16H34, C14H30, C8H16O2, C8H18, C9H19I, C10H20O, C14H30O3S, C15H24O, C15H26O, C10H18, C12H22O2, C9H14O, C18H54O9Si9, C16H48O6Si7 |
| 7 | 75%hexane + 25% chloroform | C15H24, C18H26O, C15H24O, C14H22. |
| 8 | 100%hexane | C9H12, C13H28, C14H30, C10H22, C11H24, C12H26, C8H18, C9H19I, C8H16O, C6H14, C15H18O2, C9H14O2, C15H24, C16H34, C17H36, C14H30O3S, C14H31BO, C15H26O |

Table 2: GCMS data for the fraction of gum resin of *Shorea robusta*

CONCLUSION

The *Shorea robusta* gum resin semi purified fraction (n-hexane) has the potential to work as a natural emulsifier. To know the responsible chemical moieties in n-hexane fraction, and further fractionations were helped us for GCMS analysis. All isolated fractions were subjected for GCMS analysis. This analysis data has revealed the probable chemical groups, concentration levels as per the NIST library we have considered 100 % n-hexane fraction as a reference and compared each and every sub fractions

further. Based on GCMS data 100% n-hexane fraction of the gum resin of *Shorea robusta* has the probability of the components were C9H12, C13H28, C14H30, C10H22, C11H24, C12H26, C8H18, C9H19I, C8H16O, C6H14, C15H18O2, C9H14O2, C15H24, C16H34, C17H36, C14H30O3S, C14H31BO, C15H26O. The mixture of components which were present in 100% n-hexane fraction of *Shorea robusta* was shown good emulsification properties. This study gave maximum information of the semi fractions of gum resin of *Shorea robusta*

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