

Chemical Characterization of Aerosols in a Malaysian forested area: Selai, Endau-Rompin Johor National Park

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ABSTRACT Atmospheric aerosols from Selai, Endau-Rompin Johor National Park were collected and their solvent-extractable organic compounds were converted to their trimethylsilyl derivatives and analyzed by gas chromatography-mass spectrometry. The principal organic compounds identified were sugar alcohols, triglycerides, fatty acids, hydrocarbons, sugars, and levoglucosan with percent compositions of 58%, 14%, 14%, 6%, 5%, and 3% respectively. The majority of the organic components of these forest- area aerosols is contributed by microbial sources and biomass burning.

ABSTRAK Aerosol daripada Selai, Taman Negara Johor Endau-Rompin dikumpul dan sebatian-sebatian organik terekstrak ditukarkan kepada terbitan trimetilsililnya dan dianalisis dengan menggunakan kaedah kromatografi gas-spektrometri jisim. Sebatian-sebatian organik utama yang dikenalpasti ialah alcohol gula, trigliserida, asid lemak, hidrokarbon, gula, dan levoglukosan dengan peratus masing-masing 58%, 14%, 14%, 6%, 5%, dan 3%. Sumbangan utama pecahan organik dalam aerosol kawasan hutan didapati berasal daripada sumber microbial dan pembakaran biojisim.

(Forest aerosols, Levoglucosan, Sugar alcohols, Triglycerides)

INTRODUCTION

An aerosol may be defined as a suspension of solid or liquid particles in air. The term aerosol includes both the particles and all vapor or gas phase components of air. However, the term aerosol is used here to refer to the suspended particles only. Aerosols are emitted to the atmosphere from a variety of sources including motor vehicles, industrial processes, biomass burning, cooking operations, cigarette smoke, dust from the ground and biological debris, pollen and spores. Atmospheric aerosols comprise a significant fraction of organic matter. This fraction constitutes molecular markers that act as source specific compounds [1-9].

Most of the research conducted in Malaysia has focused upon urban areas, where organic emissions are large and have dramatic effects on air quality and human health [10-13]. Nevertheless, the study of forest aerosols is of interest in order to compare their organic composition with that of urban aerosols and thus assign the sources (i.e. biogenic versus anthropogenic sources). This in turn aids in the

management of organic emissions to the atmosphere. The aim of the current study is to characterize the solvent-extractable organic matter of aerosols collected from a forested area in Malaysia (Selai, Endau-Rompin Johor National Park). The park lies in the district of Segamat, in the state of Johor. It encompasses about two third of the total area of Endau-Rompin Johor National Park. The western region of Selai, Endau-Rompin is a virgin tropical rain forest, mainly lowland mixed dipterocarp forest except Gunung Tiong, an edaphic hill forest. Within the lowland mixed dipterocarp forest there are riparian forests – consisting of plant communities adapted to river and stream edges and sandy or rocky banks, and small pockets of swamp forest near the streams. In terms of geological aspect this area consists mainly of volcanic rock (rhyolite is abundant along Sg. Selai and Gunung Tiong), plutonic rock, sedimentary rock and alluvial deposit.

EXPERIMENTAL

Forest aerosols were collected at Lubok Tapah Base Camp (02° 26' 04"N, 103° 15' 47"E), Selai,

Endau-Rompin in April 2002 using a TSP high-volume air sampler (Tisch Environmental Inc, USA) fitted with annealed (550°C for 3h) quartz fiber filters (20.3 cm × 25.4 cm, Whatman, England), and operated at a flow rate of 1.07 m³ min⁻¹. Sampling was carried out over a 24-h period.

The filter was cut into small pieces and extracted three times using ultrasonic agitation for a 15 min period each with 100 ml of dichloromethane:methanol (3:1). The extraction was carried out within the filter storage jar. The extract was then filtered using a filtration unit containing an annealed glass fiber filter for the removal of insoluble particles. The filtrate was first concentrated on a rotary evaporator and then using a stream of dry nitrogen gas to a volume of approximately 2 ml. The volume was then adjusted to 2.0 ml exactly by addition of dichloromethane.

One milliliter of the extract was derivatized by reaction with *N,O*-bis-(trimethylsilyl) (TMS)-trifluoroacetamide (BSTFA) plus 1% trimethylchlorosilane for three hours at 70°C. The derivatized extract was then concentrated to 300.0 μL and subjected to GC-MS analysis.

The analysis was carried out on a Hewlett-Packard Model 6890 gas chromatograph-mass spectrometer fitted with a fused silica capillary column coated with 5% phenylmethylsiloxane (HP-5MS, 30 m, 0.25 mm i.d, 0.25 μm film thickness). The instrument was operated at full-scan mode with an initial flow of 1.2 ml min⁻¹; nominal initial pressure: 10.4 psi; average velocity: 40 cm s⁻¹; injection mode: splitless (1

min); temperature of injector: 290°C. The GC-MS operating conditions were as follows: isothermal at 60°C for 2 min, temperature program 60-280°C at 6°C min⁻¹, held isothermal at 280°C for 30 min and using helium as the carrier gas. Data for qualitative analysis were acquired in the electron impact (EI) mode (70 eV), scanning from 50-550 mass units at 1.5 s scan⁻¹.

The identification was based on the GC-MS data (i.e. mass fragmentograms and/or mass spectra). Retention times were compared with those of available external standards. Quantification was performed from the GC profiles using the external standard method. Average response factors were calculated for each compound.

RESULTS AND DISCUSSION

In the current study, the total suspended particles (TSP) concentration is found to be 26 μg m⁻³. This value is much lower than that measured for urban Kuala Lumpur (74 μg m⁻³, TSP, 24 hours) [10] and comparable to those measured in a rural area in Eastern Mediterranean (16-37 μg m⁻³, TSP, 20-24 h) [14].

The detailed analytical results are listed in Table 1. Total ion current (TIC) chromatogram of the sample is shown in Figure 1. The organic material consisted of hydrocarbons, triglycerides, fatty acids, levoglucosan, sugars and sugar alcohols with percent compositions of 6%, 14%, 14%, 3%, 5%, and 58% respectively. The potential sources of these organic compounds will be discussed in the following sections.

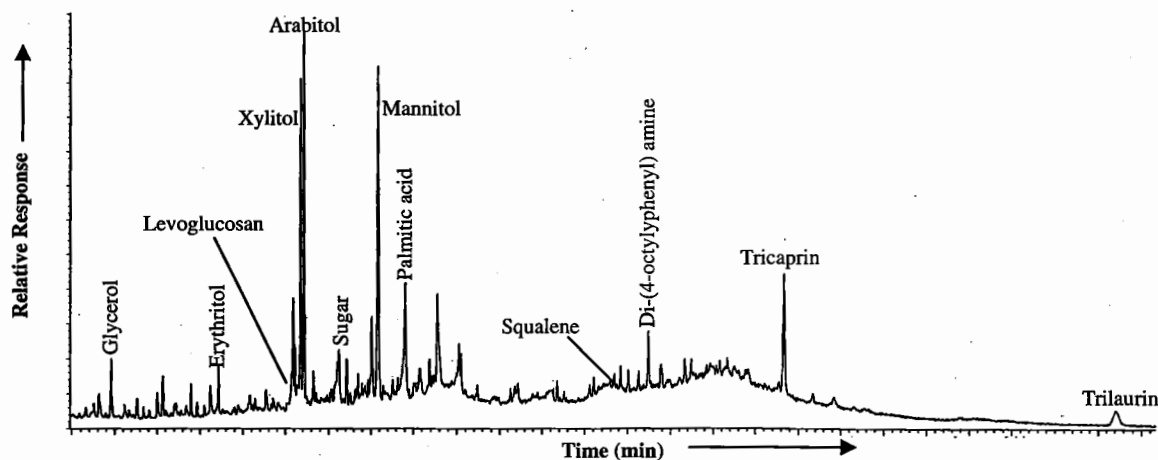


Figure 1. TIC of forest aerosol sample.

Hydrocarbons

The main n-alkanes identified are in the range of C₂₇ to C₃₁. The odd chain homologs are more abundant with carbon number maximum (C_{max}) at C₃₁ (Table 1). Plant waxes contain n-alkanes ranging from about C₂₁ to C₃₇ [15]. n-Alkanes are biosynthesized either by elongation-decarboxylation of long chain fatty acids (e.g., palmitic acid (C₁₆) is elongated in C₂ (acetate) units and subsequently decarboxylated), or by head-to-head condensation of two fatty acids, followed by decarboxylation to the ketone,

reduction to secondary alcohol, then dehydration and reduction to give n-alkane [16-18]. n-Alkanes with an even number of carbon atoms can only result from the coupling of fatty acids containing an even and an odd number of carbon atoms. Because odd carbon number fatty acids are relatively rare, virtually odd carbon number n-alkanes are predominant in plants [17]. Plant wax n-alkanes show a C_{max} in the range of C₂₅ to C₃₅, which depends on the plant species as well as the season and locality [e.g., 16, 19-22].

Table 1. Concentrations of organic compounds identified in forest aerosols

Compound	Concentration (ng m ⁻³)
Hydrocarbons	
n-Heptacosane	0.128
n-Octacosane	0.128
n-Nonacosane	0.195
n-Triacontane	0.191
n-Hentriacontane	0.276
Squalene	0.210
Triglycerides	
Tricaprin	2.016
Trilaurin	0.768
Fatty acids	
Lauric acid	0.281
Myristic acid	0.729
Palmitic acid	1.677
Anhydrosugars	
Levogluconane	0.670
Sugars	
Sugars (total)	1.034
Sugar alcohols	
Glycerol	0.676
Erythritol	0.431
Xylitol	2.814
Arabitol	3.109
Mannitol	4.254
Contamination	
bis-(octyl pheny) amine	0.570

Squalene (Structure I, all cited structures are listed in the Appendix) is also detected in this sample (Table 1). This compound is absent from the surface layer of leaves but present in significant amounts in human skin [23]. Also, palm oil has squalene content ranging from 0.4 to 0.9 mg g⁻¹ of oil [24]. In addition, squalene had been detected in yeasts and fungal lipids [25]. Thus the presence of squalene may be attributed to cooking operations utilizing palm oil or from microorganisms which are hosted by plants and due to wind-induced mechanical shear and rubbing of leaves against each other, such microorganisms become airborne and contribute to the organic content of the aerosols [4].

Wax esters

The two major wax esters identified in the sample are tricaprins and trilaurins. Although triglycerides (triesters or triacylglycerols) are ubiquitous in the internal tissues of mammals, their occurrence in surface lipids is only certain in human [26]. Also, they have been found in cuticular lipids in some insects and birds [27, 28]. The fat in uncooked meat occurs as triglycerides, which consist of three fatty acids esterified to a glycerol backbone. However, during the cooking process, triglycerides can be hydrolyzed or thermally oxidized, yielding free glycerol, mono- and diglycerides, and free fatty acids [29] and the precursor compounds (i.e. triglycerides) are not

present in aerosols originated from meat cooking operations [1, 29]. Many plants have triglycerides in their lipids, which are important energy storage molecules. It should be noted that these common lipids are not present in epicuticular waxes but are internal constituents of the plants. They have a very low vapor pressure and thus directly volatilized during burning of plants [30, 31]. From the above discussion, the presence of tricaprins and trilaurins in the air sample (Table 1, Structures II and III, respectively, Figure 2 shows the mass spectra of tricaprins) can be related to biomass burning origin and/or human/birds/insects contributions of their surface lipids to the aerosols.

Fatty acids

Plant wax n-alkanoic acids are generally much longer (> C₂₀) than those found in internal lipids [15]. These n-fatty acids are biosynthesized by elongation of low molecular weight homologues resulting in predominantly even carbon number n-alkanoic acids [18]. Microorganisms such as bacteria, fungi and algae favor production of low molecular weight n-alkanoic acids (< C₂₀) [32]. The most abundant fatty acid detected in the current study is palmitic acid (C₁₆) (Table 1) which maybe related to microbial sources, although this acid is found in some plants [e.g., 2]. Meat cooking operations [1], Cigarette smoke [6], and biomass burning [33, 34] may also be possible sources of this compound and other lower molecular weight fatty acids.

Anhydrosugars, sugars, and sugar alcohols

Cellulose (Carbohydrate biopolymer) decomposes on heating or on exposure to an ignition source by two alternative pathways. The first, which dominates at temperatures < 300°C, involves depolymerization, water elimination, fragmentation and oxidation leading finally to char formation. The second pathway occurs at temperatures > 300°C and involves bond cleavage by transglycosylation, fission and disproportionation reactions yielding tarry

anhydrosugars and volatile products [31]. Levoglucosan (1,6-anhydro-β-D-glucopyranose, Structure IV) which is produced by the second pathway has been detected in aerosols from vegetation burning [33-36]. This compound is stable in the atmosphere, showing no decay over 8 h exposure to ambient conditions and sunlight. Incidentally, other carbohydrate polymers (e.g., starch) also produce levoglucosan upon thermal alteration. However, the temperatures attained in cooking, baking or toasting are not sufficient to pyrolyze carbohydrates to levoglucosan and thus these lower temperature processes are not a significant source [31]. Therefore, the presence of levoglucosan in the studied sample (Table 1, Figure 3(a)) is interpreted to be of vegetation burning origin. The mass spectrum of levoglucosan-TMS ether exhibits only a small molecular ion (m/z 378) with fragments due to loss of CH₃ (m/z 363), CH₃Si (m/z 333), C₆H₁₇OSi₂ (m/z 217) and C₇H₁₈OSi₂ (m/z 204, base peak) [35].

Sugars and sugar alcohols (also known as alditols or polyols, Structures VI-IX) are also detected in the studied sample (Table 1, Figures 3(a) and (b)). The fragmentation of TMS ethers of sugars generates the ions m/z 191 [(CH₃)₃SiOCH=OSi(CH₃)₃]⁺, 204 [(CH₃)₃SiOCH=CHOSi(CH₃)₃]⁺ and 217 [(CH₃)₃SiOCH=CH-CH=OSi(CH₃)₃]⁺. The ions m/z 205 [(CH₃)₃SiOCH₂-CHOSi(CH₃)₃]⁺, 217 [(CH₃)₃SiOCH=CH-CH=OSi(CH₃)₃]⁺ and 319 [(CH₃)₃SiOCH=CH-CH(OSi(CH₃)₃)-CHOSi(CH₃)₃]⁺ are characteristics of the mass spectra of sugar alcohols. It is possible that a number of these compounds are primary products of combustion, formed either through direct volatilization from plant matter or as products of the breakdown of polysaccharides (i.e. cellulose) [37]. Some of the sugars may also be produced by hydrolysis of the corresponding anhydrosugars under the acidic atmospheric conditions created by biomass burning [37].

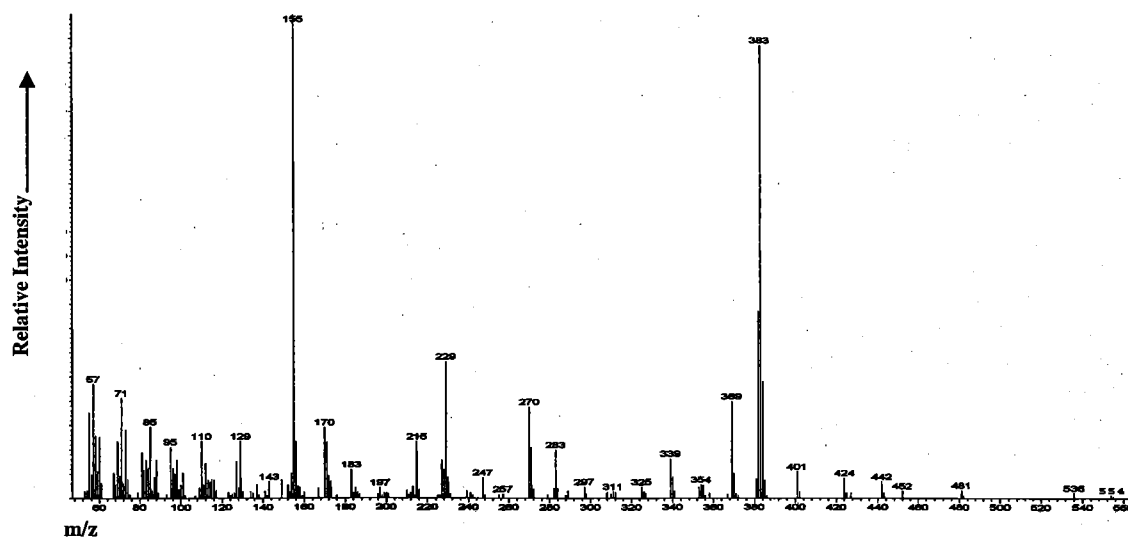


Figure 2. Mass spectra of tricaprins

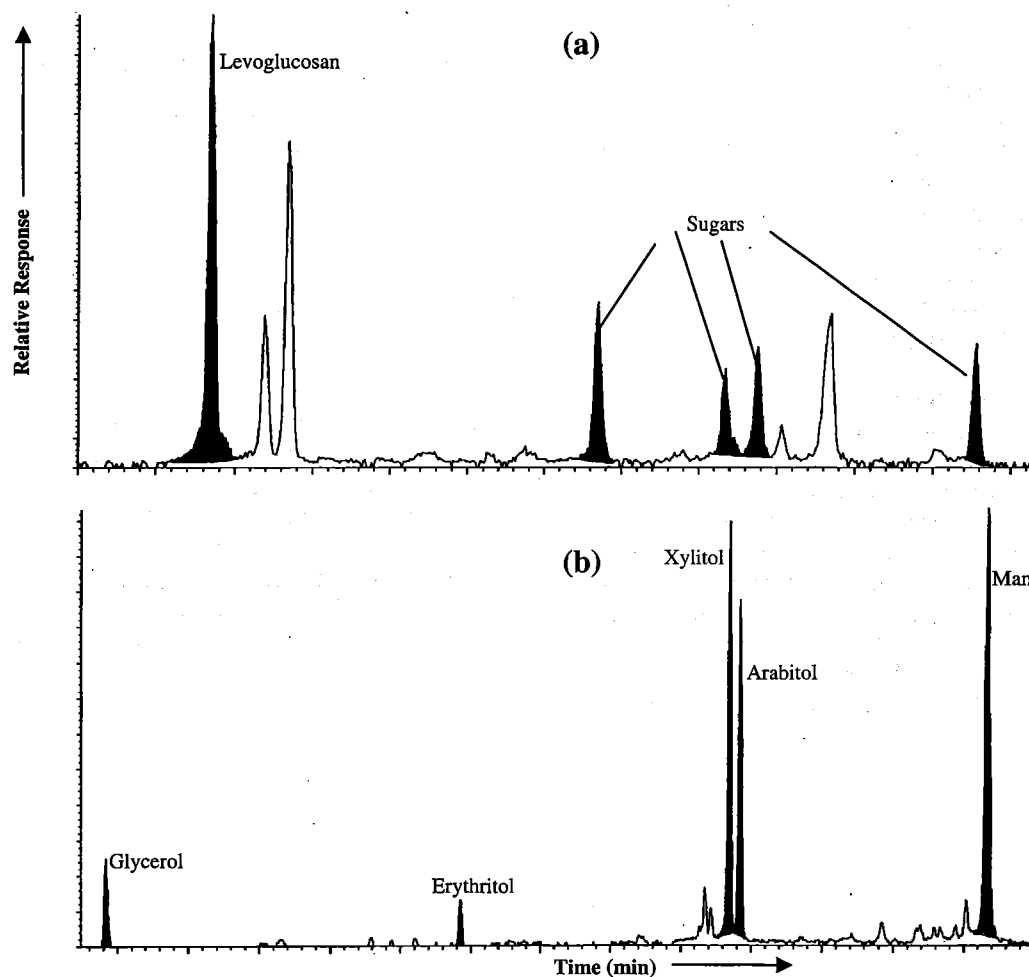


Figure 3. (a) m/z 204 Mass fragmentogram typical of anhydrosugars and sugars and (b) m/z 205 mass fragmentogram typical of sugar alcohols.

Graham *et al.* [37] and Carvalho *et al.* [38] have suggested that in addition to biomass burning as a source of sugars and sugar alcohols a further, in some cases dominant, source of these compounds may be suspended biogenic detritus which forms part of the natural background aerosol material (plant detritus, airborne microbes comprised of bacteria, viruses, spores of lichen and fungi, small algae, and protozoan cysts). Indeed, sugar alcohols are common in bacteria, fungi, and lower plants with mannitol and arabitol being the most widespread in fungi despite that erythritol, ribitol and glycerol are also usually found in fungi [37, 38 and references cited therein]. Based on electron microprobe analyses performed on filter samples collected in the Amazon Basin, Artaxo *et al.* [39, 40] had confirmed that there was an enormous diversity of biogenic particles including pollen, fungal spores, bacteria, algae, fragments of leaves, and excrement and fragments of insects. Also, Graham *et al.* [37] showed that sugars and sugar alcohols were the major species identified by the GC-MS method in the absence of biomass burning.

Generally, the concentrations of sugars and sugar alcohols are relatively lower compared to that of levoglucosan [e.g.; ref. 37 and 41]. However, in the studied sample, the concentrations of sugar alcohols are much higher than that of levoglucosan (Table 1, Figure 1) implying that biogenical sources (e.g., fungal fragments present in the forest aerosols) are more abundant than biomass burning. This is supported by the fact that the air sample was collected at ground level under the forest canopy where the production of coarse biogenic aerosols is higher [40]. Fungi, for example, exist as a biogenic aerosol in the supermicrometric range ($D_p > 2 \mu\text{m}$) [38] and thus more concentrated in and under the forest canopy. In contrast, levoglucosan has been detected in fine particles ($D_p < 2 \mu\text{m}$) resulting

from biomass burning [41] which are higher over the height of the forest [40].

Glycerol (Structure V) is also present in the studied sample. This compound may be originated from a variety of sources including biomass burning [37], microorganisms (e.g., fungi) [38], meat cooking operations [29] and cigarette smoke [42]. However, the last two sources are expected not to have significant contribution in a forested area.

Contamination

Di-(4-octylphenyl) amine (Structure X) is detected at a concentration level comparable to other organic compounds found in the same sample. This compound is an antioxidant lubricant additive [43] and might have been introduced to the sample from the power generator used to supply electricity to the high-volume air sampler.

CONCLUSION

Forest aerosol sample collected from Endau-Rompin, Malaysia has been characterized for its organic contents using GC-MS. As shown in Figure 4, Sugar alcohols were the most abundant compounds having 58% of the total identified organics. Triglycerides, fatty acids, hydrocarbons, sugars, and levoglucosan had 14%, 14%, 6%, 5%, and 3% respectively.

Two major sources of these organic compounds were suggested; biomass burning and microbial sources (e.g., fungi). Sugar alcohols were dominantly originated from microbial sources, whereas levoglucosan and triglycerides were chiefly originated from vegetation burning. Therefore, the contribution of microbial sources to the forest aerosols is much higher than that of biomass burning (58% vs. 17%).

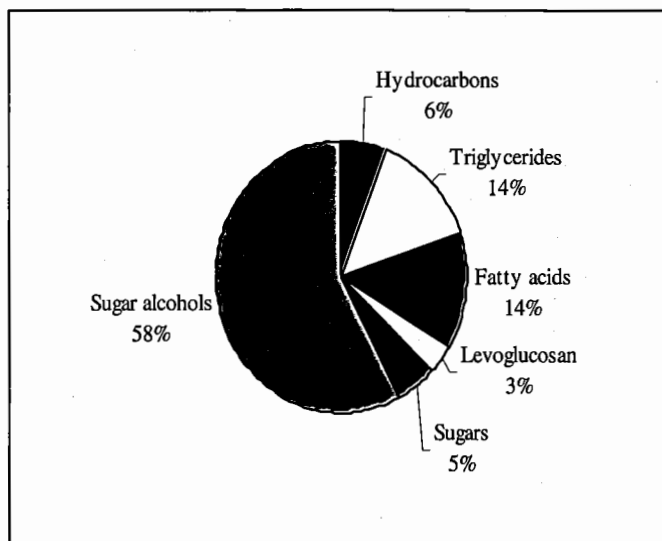


Figure 4. Composition of organic compounds identified by GC-MS for forest aerosols sample

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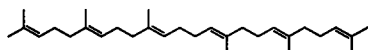
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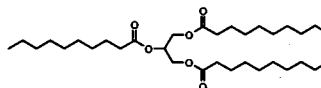
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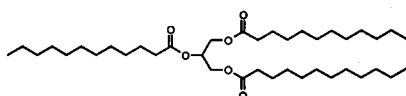
APPENDIX: Chemical Structures Cited



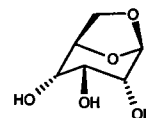
I. Squalene



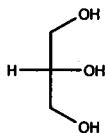
II. Tricaprin



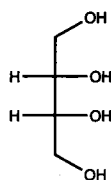
III. Trilaurin



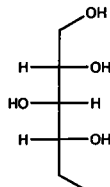
IV. Levoglucosan



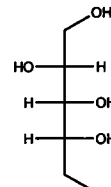
V. Glycerol



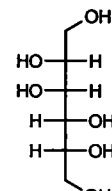
VI. Erythritol



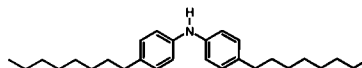
VII. Xylitol



VIII. Arabitol



IX. Mannitol



X. Di-(4-octylphenyl) amine