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Short Communication

CHARACTERIZATION OF WOOL OF THALLI SHEEP BY GAS CHROMATOGRAPHY – MASS SPECTROMETER (GC-MS)

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ABSTRACT

Organic compounds present in wool wax have several applications. They can be used either direct or as raw materials to synthesize many important products. In the present study, these compounds were extracted from sheep wool with n-hexane, benzene and chloroform using a Soxhlet apparatus. The extracts were characterized using gas chromatograph coupled to a mass spectrometer. Chloroform and n-hexane infusions were found to contain 3-butyn-1-ol, cyclopropene, 2-methyl octane, 2-methyldecane, p-xylene, benzonitrile and ethyl benzene while extract of benzene own 2, 4-dimethyl hexane, 1, 2-dimethyl cyclohexane, 1-ethyl 3-methyl cyclopentane, octane, 2-methyl octane, n-propyl cyclopentane, ethyl cyclohexane, 1, 3-dimethyl benzene, o-xylene, nonane, 1-pentanol, 1, 2, 3-trimethyl benzene, decane, 5-ethyl-2-methyl heptane, 1, 6-diol-2, 7-octadiene, 5-(2-methylpropyl)-nonane, pentyl cyclohexane, 1, 7, 7-trimethyl-2-vinyl-bicyclo-hept-2-ene and 2, 3, 5, 8-tetramethyl decane.

Keywords: Sheep wool, extraction; benzene, chloroform, n-hexane, soxhlet.

INTRODUCTION

Sheep wool is known as a strong fiber. It can bend on itself 20,000 times without breaking, which is high compared with other fibers such as cotton (3,200), silk (1,800) and rayon (75) (Nostran, 2006). Wool, which usually contains up to 20 amino acids, has a complex chemical structure, which accounts for its unique character as a fiber (Ludecke and Invanvszky, 1958). Efficiency in handling body moisture in both hot and cold conditions results from the porous structure of wool creating millions of miniature air pockets which help to regulate temperature and humidity. Sheep wool is covered in a protective layer of lanolin, a waxy substance with a faint characteristic odor which is a mixture of animal fats. Lanolin is formed by the oleaginous glands of sheep and is vital for greasing the wool and preserving the skin (Eychenne *et al*., 2001). Lanolin is removed in a pretreatment process and is widely used in the pharmaceutical and cosmetic industries in the production of moisturizers and body lotions.

A numbers of organic compounds are present in lanolin which can be used either directly or as a starting material for many important products. Wool is often considered as a potential internal source of volatile organic compounds (Lisovac and Shooter, 2003). Factors such as diet, sheep breed, living conditions, pregnancy, lactation, color and texture affect emission of volatiles from sheep wool. In addition, different types of bacterial and fungal flora use wool as their source of nutrition (Burrell, 1990; Gochel *et al*., 1990). Although, fleece wool may have some antibacterial properties (Meyer *et al*., 2001). The Maillard reaction between amino acids and reducing sugars, or amino acids and lipid deterioration products are two important reactions in the formation of volatile organic compounds of wool (Farmer, 1996).

The amount of wool grown per sheep is a function of the geographical area, the wool follicle density and volume of fiber per follicle (Adelson *et al*., 2004). Sheep wool is composed of fibrous and hard α-keratins (Marshall *et al*., 1991) and is metabolically dead after leaving the epidermis (Raab *et al*., 2002). Wool consists of three basic components; the cuticle, the cortex and medulla. The internal cortical cells have long polyhedral spindle-shaped structures (Jones, 2001). The natural coloring in the fibers is provided by the pigment melanin (Feughelman, 2001).

The present study describes the extraction of various organic compounds from wool fibers of Thalli sheep (a local breed) and the evaluation of the performance of n-hexane, chloroform and benzene as solvent for the extraction of these compounds using a Soxhlet apparatus. The resulting extracts were characterized by using a gas chromatograph coupled with the mass spectrometer.

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MATERIALS AND METHODS

Wool sampling

Wool samples from six months old male Thalli sheep were collected from the local market Lahore, Pakistan. The samples were thoroughly washed with water to remove contaminating dust, grass and other exogenous materials. The samples were dried in an oven at 50°C and cut into small strands with a sharp knife.

Extraction with Soxhlet extractor

A Soxhlet extractor with cellulose thimble was used for the solvent extraction. The sheep wool (8.25g) was placed in the thimble and 500mL of pure solvent put into the round bottomed flask. The three components of the extractor were put together with flask in an isomantle fitted with a regulator and cold water (20°C) running continuously in the condenser. The temperature was adjusted so that the solvent boiled. The sample was extracted for 10hours. When extraction was complete the extract was washed from the flask with the respective solvent. The solvent was removed by simple distillation.

Characterization

The components extracted from the sheep wool were characterized using gas chromatograph coupled with the mass spectrometer figures 1-3. A Shimadzu (QP-2010 GC-MS, Japan) equipped with column (HP DB5 Agilent, length: 30m, internal diameter: 0.25mm, film thickness: 0.25mm), was used. The flow rate of the helium carrier gas was adjusted to 1mL/min. The GC-MS operating conditions were: injection temperature, 280°C; detector temperature, 200°C; initial oven temperature, 30°C; electron impact, 70 eV; mass range, 4-1020m/z and ion source temperature, 200°C.

RESULTS AND DISCUSSION

Volatile organic compounds, most of which were aliphatic hydrocarbons (saturated and unsaturated), aromatic hydrocarbons, amines and alcohols, were detected and identified in sheep wool using GC-MS equipped with library NIST 127/47. The retention time, molecular formula and weights of various compounds acquired by extraction with n-hexane, chloroform and benzene are shown in tables 1-3 respectively.

The n-hexane extract contains 3-butynyl-1-ol, 2-methyloctane, cycloprene, and 2-methyldecane. The chloroform extract contains 1, 3-cyclopent-di-ene, ethyl benzene, 3-butynyl-1-ol, p-xylene, 1-benzyloxy -5-diethylamino-2, 4-dinitro benzene and 4-benzyloxy benzyliden amino benzonitrile. It appears that benzene is the most suitable solvent because of the number of compounds extracted, but the choice of solvent is also governed by the compound required. The benzene extract contains 2, 4-dimethyl hexane, 1, 2-dimethyl cyclohexane, 1-ethyl 3-methyl cyclopentane, octane, 2-methyl octane, n-propyl cyclopentane, ethyl cyclohexane, 1, 3-dimethyl benzene, o-xylene, nonane, 1-pentanol, 1, 2, 3-trimethyl benzene, decane, 5-ethyl-2-methyl heptane, 1, 6-diol-2, 7-octadiene, 5-(2-methylpropyl)-nonane, pentyl cyclohexane, 1, 7, 7-trimethyl-2-vinyl-bicyclo-hept-2-ene and 2, 3, 5, 8-tetramethyl decane.

Table 1. Different constituents extracted by n-hexane.

Name	Mol. Formula	Mol. Weight	Retention Time (min)
3-Butynyl-1-ol	C_4H_6O	70	10.850
2-Methyl octane	C_9H_{20}	128	10.850
Cycloprene	C_3H_4	40	12.908
2-Methyl decane	$C_{11}H_{24}$	156	14.375

It is not suggested that each type of wool will contain the specific volatile organic compounds found here. The composition of the extracts depends on factors such as sheep breed, age, diet and habitat. Additionally, detection of compounds depends on the sensitivity of the current analytical technology. Gas chromatography-mass spectrometry (GC-MS) is a combination method which is used to identify different volatile substances in a sample. The use of a mass spectrometer as the detector in gas chromatography was developed during the 1950s (Gohlke and McLafferty, 1959). Only compounds with vapor pressures exceeding about 10^{-10} torr can be analyzed by (GC-MS). Many compounds with lower pressures can be analyzed by this method, but they must first chemically derivatized to a form with required vapour pressure. Deciding positional substitution on aromatic rings is often difficult. Certain isomeric compounds cannot be

Table 2. Different constituents extracted by chloroform.

Name	Mol. Formula	Mol. Weight	Retention Time(min)
1, 3-Cyclopent-di-ene	C_8H_{10}	106	4.400
Ethyl benzene	C_8H_{10}	106	4.400
3-Butynyl-1-ol	C_4H_6O	70	4.867
p-Xylene	C_8H_{10}	106	4.233
1-Benzyloxy -5-diethylamino-2, 4-dinitro benzene	$C_{17}H_{19}N_3O_5$	345	4.233
4-Benzyloxy benzyliden amino benzonitrile	$C_{21}H_{16}N_{20}$	312	4.233

Table 3. Different constituents extracted by benzene.

Name	Mol. Formula	Mol. Weight	Retention Time (min)
2, 4-Dimethyl hexane	C_8H_{18}	114	3.208
1, 2-Dimethyl cyclohexane	C_8H_{16}	112	3.292
1, 4-Dimethyl cyclohexane	C_8H_{16}	112	3.831
1-Ethyl 3-methyl cyclopentane	C_8H_{16}	112	3.158
Octane	C_8H_{18}	114	3.208
2-Methyl octane	C_9H_{20}	128	3.625
n-Propyl cyclopentane	C_8H_{16}	112	3.733
Ethyl cyclohexane	C_8H_{16}	112	3.808
1, 3-Dimethyl benzene	C_8H_{10}	106	4.408
o-Xylene	C_8H_{10}	106	4.875
Nonane	C_9H_{20}	128	4.967
1-Pentanol	$C_9H_{20}O$	144	5.775
1, 2, 3-Trimethyl benzene	C_9H_{12}	120	7.467
Decane	$C_{10}H_{22}$	142	8.275
5-Ethyl-2-methyl heptanes	$C_{10}H_{22}$	142	8.275
1, 6-Diol-2, 7-octadiene	$C_{10}H_{18}O_2$	170	11.417
5-(2-Methylpropyl)-nonane	$C_{13}H_{28}$	184	11.750
Pentyl cyclohexane	$C_{11}H_{22}$	154	12.117
1, 7, 7-Trimethyl-2-vinyl-bicyclo-hept-2-ene	$C_{12}H_{18}$	162	12.908
2, 3, 5, 8-Tetramethyl decane	$C_{12}H_{30}$	198	13.092

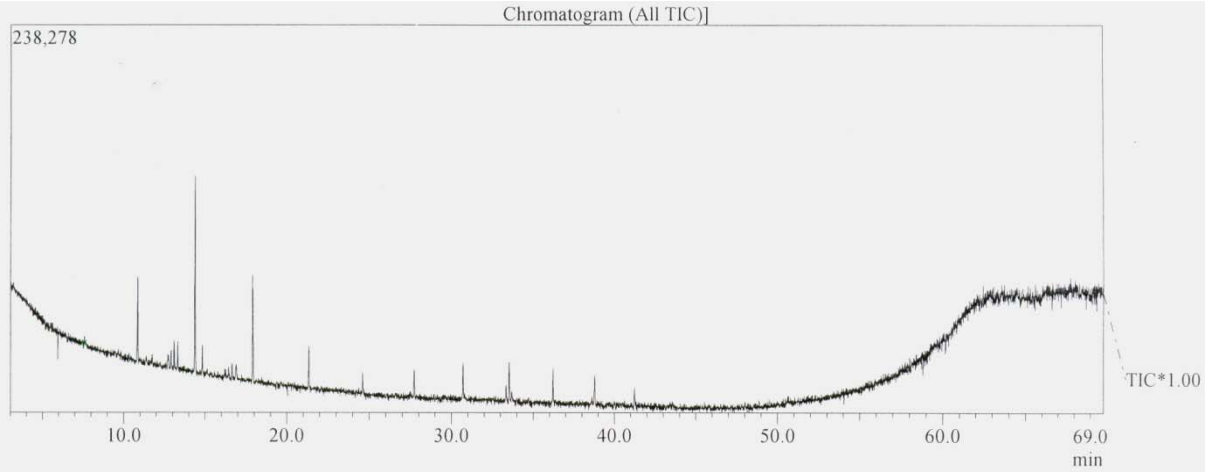

Fig.1. Extraction of sheep wool with n-hexane.

distinguished by mass spectrometry, but can often be separated chromatographically (Hites, 1997). No sulphur compound was detected in the present study, even though such substances are responsible for the smell of raw sheep wool.

CONCLUSION

In the present study, different organic compounds have been extracted from sheep wool. These compounds were characterized by GC-MS. The study assessed the performance of benzene, n-hexane and chloroform as solvent. Benzene extracted more compounds compared to the other solvents.

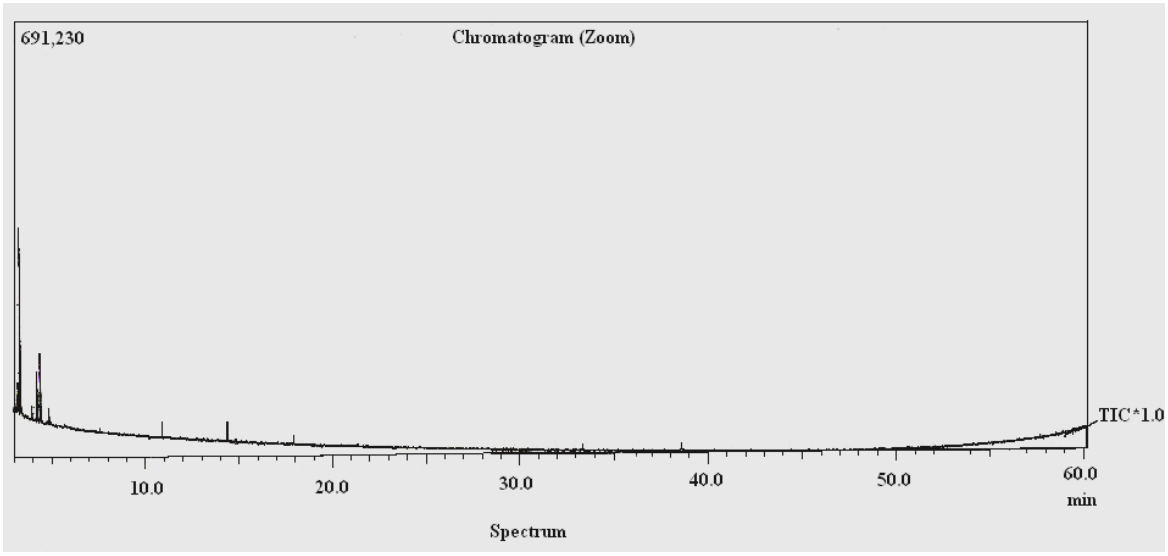

Fig. 2. Extraction of sheep wool with chloroform.

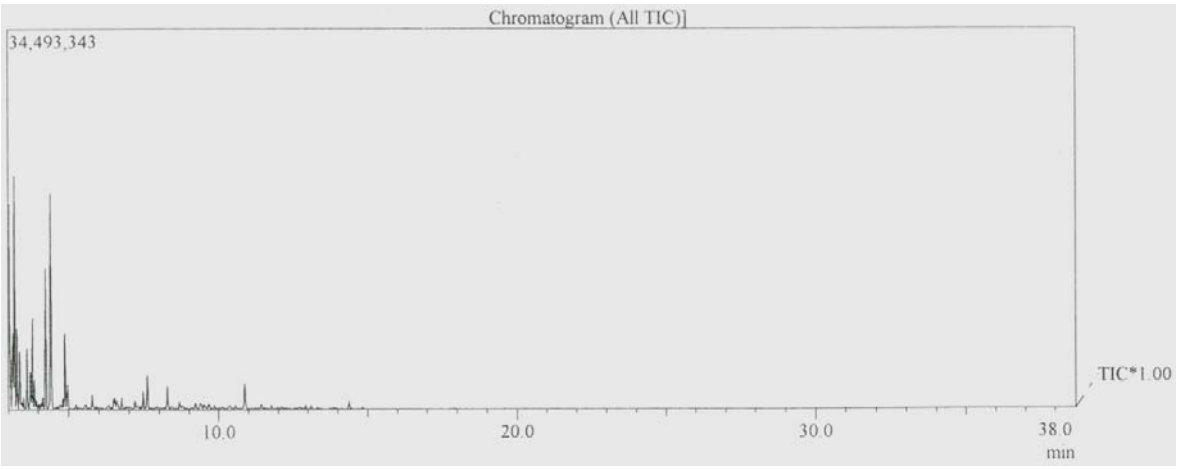

Fig. 3. Extraction of sheep wool with benzene.

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