

GC–MS Investigation of Volatile Organic Compounds in Cosmetics Manufactured in South India

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Abstract

The volatile organic compounds (VOCs) in cosmetics pose a serious threat to the environment and their exposure causes a variety of ailments in human beings. The present work analyses systematically the presence of VOCs in samples of cosmetic products available in open market such as, aftershave lotion, face cream, hair dye, lipsticks, perfumes, shampoos and talcum powders by GC–MS. The investigation revealed the prevalence of more than 158 hazardous volatile organic compounds (VOCs) out of 243 organic compounds in 20 samples of the cosmetic products. The detected VOCs fall into six major groups *viz.*, alcohols, esters, ethers, carboxylic acids and amides. Among all the VOCs detected, diethyl phthalate is found to be present in majority of the samples, which could cause mild eye and skin irritations and affect the central nervous system depending on the dosage. The samples were also found to contain certain fatty acid methyl esters. The study suggests proper instructions on the labels of such products to warn the customers. Further, finding non-toxic alternatives to existing additives has also been emphasized.

Introduction

The emergence of cultured lifestyle has forced the population all over the world to use cosmetic products to keep oneself presentable. Cosmetic products with pleasant odors play an important part in day-to-day life (Kalicinin and Velimirovic 2016; Buckle 2001; Chu and Downes 2000). The first evidence of human usage of perfumes dates back to three thousand years, when Egyptians used plants, gums, and resins in religious rites (Lopez- Sampson and Page 2018). There is a direct relationship between the growth of fashion and the production of personal care products (Beausoleil 1992). Cosmetic products have attracted both the sexes and their usage gives them confidence in their personal and public life. Face creams and talcum powders, once thought to be a sole possession of women, are now available for men as well. The recent popular notion is that a cultured person is that one who uses the maximum possible personal care products. The chemicals present in these products come into contact with and remain in skin, hair, nail, lip, or any part of the body for a long time (Danish Khan and Alum 2019). Thus, a new area of research in the health sector has arisen to study the consequences of some harmful products on human health (Hagerman et al. 1997).

Volatile organic compounds are a large and diverse group of organic compounds that are prone to be volatile at room temperature (Boeglin et al. 2006). Both anthropogenic processes and natural phenomena are responsible for the release of VOCs. The manifold ill effects of such compounds even at trace amounts make their detection a necessity (Nazaroff and Weschler 2004; Sarigiannis et al. 2011; Son et al. 2003). Diagnosis of the severity of air pollution due to VOCs and the threat they pose on the well-being of humans finds utmost importance. (Singer et al. 2006).

Bridges et al., reported that hanky perfumes, body sprays and deodorants comprise of a spectrum of hazardous chemical that can target the human respiratory system (Bridges 2002). The VOCs used in cosmetics have been grouped into several classes based on their chemical nature and their biological interventions in the functioning of the human body (Rahman and Kim 2014). To illustrate the harmful effects of VOCs, the ban on attars in the wake of reported allergens in Japan during the 1980s can be cited (Peak et al. 2006).

Among the variety of chemicals used in the making of cosmetics like shampoos, aftershave lotions, beauty creams, hair dyes, lipsticks, deodorants and fragrance powders, volatile organic compounds (VOCs) have

attracted the attention of researchers as invariably all of the beauty products are scented using hundreds of aromatic chemicals to give the characteristic aroma of the product (Fortineau 2004; Schwab et al. 2008; Liu and Little 2012).

Azeez et al have reported the presence of formaldehyde, toluene, trichloroethene and tetrachloroethene (volatile organic compounds) present in Nigerian-made cosmetics such as, medicated powder, talcum powder, lipstick and nail paint (Azeez et al. 2013). Hair care products including shampoo available in UK have been assessed for VOCs by selected ion flow-tube mass spectrometry (Yeoman et al. 2020). Hadei et al have reported the presence of various toxic VOCs at various concentrations in hair care products including hair dyes (Hadei et al. 2018). Recently, fragranced consumer products have been examined for the presence of hazardous VOCs and their effect on human health was studied (Steinemann 2021).

Intrigued by the above studies carried out internationally, the present investigation attempts to detect the hazardous volatile organic chemicals released and other major chemical compounds from various cosmetic products manufactured and marketed in South India.

Materials And Methods

The samples of cosmetic products, *viz.*, aftershave lotion, face cream, hair dye, lipstick, perfume, shampoo and talcum powder manufactured in south India were collected from the commercial vendors in Coimbatore, Tamil Nadu, India. The samples were stored as mentioned in the prescribed instructions after ensuring that the shelf life lies well within the expiry period. The details of the type and number of samples (of various brands) are shown in Table 1. All of the samples were analyzed by GC-MS for the presence of VOCs and other organic compounds. Sample preparation was done using AR grade chemicals (BDH, India). The talcum powder and powder hair dye samples were easy to handle and weighed using suitable plastic containers. The samples of aftershave lotion, face cream and shampoo were weighed using non-stick silicone wax containers. The lipstick samples were collected for weighing by scratching with a clean blade. The perfume samples were collected carefully in glass containers provided with airtight lids.

Table 1
Classes of cosmetic varieties

Cosmetic variety	No. of samples
Aftershave lotion	2
Face cream	3
Hair dye	2
Lipstick	2
Perfume	5
Shampoo	3
Talcum powder	3

The VOCs present in the test samples were extracted from adopting a well-established procedure (Rastogi and Jensen 1995). In brief, about 1 g of each cosmetic sample was taken in a 10-mL standard measuring flask and filled up with methanol. The mixture was shaken gently and then heated at 60°C for 10 min. The sample solutions were subsequently analyzed for volatile compounds by a Shimadzu QP 2010 GC–MS instrument equipped with a split/splitless injector and an Rtix 5 MS column. The carrier gas at a linear velocity of 36.8 cm/s using the line-of-sight interface was kept at 260°C and the ion-source temperature was set to 250°C. The prepared samples were manually injected and the rate of injection was maintained at 1.0 mL/min. The flow rate was maintained in splitless mode, the column oven temperature was maintained at 80°C and the injection temperature was maintained at 250°C. Mass spectra were obtained with an electron impact of 70 eV in the m/z range 50–800.

Results And Discussion

The exposure to VOCs from cosmetics deems a deep attention. Thus, the current investigation attempts to evaluate the presence of various VOCs in cosmetic products that can have direct contact with human body. The 20 cosmetic samples included two samples of aftershave lotions, three samples of face creams, two samples of hair dyes, two samples of lipsticks, five samples of body spray perfumes, three samples of shampoos and three samples of talcum powders samples of various brands. From the GC-MS studies, as much as 158 hazardous volatile organic compounds (VOCs) out of 243 organic compounds were identified in 20 samples (Table 2). From the data, it can be observed that maximum number of VOCs are found in perfumes (61), followed by shampoos (33). The aftershave lotions, face creams and talcum powders contain 19, 18 and 16 VOCs, respectively. Lipsticks (6) and hair dyes (5) were found to have the least number of VOCs. However, the work focuses only on VOCs and fatty acid methyl esters (FAMEs) present in more than two cosmetic samples. In this way, Tables 3 and 4 show 21 VOCs present in the products. The VOCs comprise of alcohols, esters, ethers, acids and amides. Similarly, Tables 5 and 6 show the important FAMEs present in the products. Of the 21 listed VOCs, perfume and shampoo samples have 9 VOCs and the hair dye samples have only one VOC, *viz.*, dasycarpidan-1-methanol, acetate (ester). The lipstick samples contain two VOCs namely, 1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[g]-2-benzopyran and diethyl phthalate. Likewise, it should be underlined that the compound diethyl phthalate was found in a maximum of 3 samples of the selected cosmetic products. The following paragraphs discuss the presence of important VOCs and FAMEs present in the sample and their health hazards.

Table 2
Sample code of cosmetics and number of compounds identified

S. No	Sample	Sample code	No. of compounds identified	No. of VOCs
1	Aftershave lotion	A-1	17	12
2	Aftershave lotion	A-2	13	7
3	Face cream	F-1	11	5
4	Face cream	F-2	13	10
5	Face cream	F-3	11	3
6	Hair dye	H-1	02	1
7	Hair dye	H-2	08	4
8	Lipstick	L-1	08	4
9	Lipstick	L-2	06	2
10	Perfume	P-1	13	9
11	Perfume	P-2	11	6
12	Perfume	P-3	23	17
13	Perfume	P-4	17	15
14	Perfume	P-5	18	14
15	Shampoo	S-1	19	15
16	Shampoo	S-2	16	12
17	Shampoo	S-3	10	6
18	Talcum powder	T-1	11	6
19	Talcum powder	T-2	09	8
20	Talcum powder	T-3	07	2

Table 3

VOCs present (peak area percentage) in aftershave lotion, face cream, hair dye, and lipstick samples

S. No	VOCs	A-1	A-2	F-1	F-2	F-3	H-1	H-2	L-1	L-2
1	1-Dodecanol C ₁₂ H ₂₆ O									
2	1-Tetradecanol C ₁₄ H ₃₀ O									
3	2,7-Dimethyl-2,7-octanediol C ₁₀ H ₂₂ O ₂		2.4							
4	3,7-Dimethyl-6-octen-1-ol C ₁₀ H ₂₀ O									
5	Benzyl alcohol C ₇ H ₈ O	6.5								
6	2-(Dodecyloxy) ethanol C ₁₄ H ₃₀ O ₂									
7	4- <i>tert</i> -Butylcyclohexyl acetate C ₁₂ H ₂₂ O ₂			3.7	3.6					
8	Butylated hydroxytoluene C ₁₅ H ₂₄ O			2.4						
9	1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[<i>g</i>]-2-benzopyran, C ₁₈ H ₂₆ O	9.6							9.6	6.1
10	Diethyl phthalate C ₁₂ H ₁₄ O ₄	4.5	8						5.4	
11	Diethylene glycol monododecyl ether C ₁₆ H ₃₄ O ₃									
12	D-limonene C ₁₀ H ₁₆									
13	10-Methylundecanoic acid methyl ester C ₁₃ H ₂₆ O ₂									
14	Thiopropionamide C ₃ H ₇ NS.									
15	Triethylene glycol monododecyl ether C ₁₈ H ₃₈ O ₄									
16	Indan-1,3-diol mono acetate C ₁₁ H ₁₂ O ₃									
17	2-(Phenylmethylene) octanal C ₁₅ H ₂₀ O									
18	Patchouli alcohol C ₁₅ H ₂₆ O		4							
19	(3,8,8-Trimethyl-1,2,3,4,5,6,7,8-octahydro-2-naphthalenyl)methyl acetate C ₁₆ H ₂₆ O ₂				5.4					

S. No	VOCs	A-1	A-2	F-1	F-2	F-3	H-1	H-2	L-1	L-2
20	1,2,3,4,7,7a-Hexahydro-1,4,4,5-tetramethyl-6-acetyl-1,3a-ethano-3aH-indene C ₁₇ H ₂₆ O									
21	Dasycarpidan-1-methanol, acetate (ester) C ₂₀ H ₂₆ N ₂ O ₂							11		

Table 4
VOCs present (peak area percentage) in perfume, shampoo and talcum powder samples

S. No	VOCs	P-1	P-2	P-3	P-4	P-5	S-1	S-2	S-3	T-1	T-2	T-3
1	1-Dodecanol C ₁₂ H ₂₆ O						7.5	7.1	10.2			
2	1-Tetradecanol C ₁₄ H ₃₀ O						2.9	6.6				
3	2,7-Dimethyl-2,7-octanediol C ₁₀ H ₂₂ O ₂									8.2		
4	3,7-Dimethyl-6-octen-1-ol C ₁₀ H ₂₀ O	2.8				3.7						
5	Benzyl alcohol C ₇ H ₈ O			4								
6	2-(Dodecyloxy) ethanol C ₁₄ H ₃₀ O ₂						9.6	7.1	8.33			
7	4- <i>tert</i> -Butylcyclohexyl acetate C ₁₂ H ₂₂ O ₂									7.3		
8	Butylated hydroxytoluene C ₁₅ H ₂₄ O											7.1
9	1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[<i>g</i>]-2- benzopyran, C ₁₈ H ₂₆ O											
10	Diethyl phthalate C ₁₂ H ₁₄ O ₄	6.4		6		13						
11	Diethylene glycol monododecyl ether C ₁₆ H ₃₄ O ₃						7.1	7.1	9.26			
12	D-limonene C ₁₀ H ₁₆			4	5.1	5.1						
13	10-Methylundecanoic acid methyl ester C ₁₃ H ₂₆ O ₂				5		4.5					
14	Thiopropionamide C ₃ H ₇ NS				3.6	3.7						
15	Triethylene glycol monododecyl ether C ₁₈ H ₃₈ O ₄						7.9	7.1				
16	Indan-1,3-diol mono acetate C ₁₁ H ₁₂ O ₃	7.6					2					
17	2-(Phenylmethylene) octanal C ₁₅ H ₂₀ O						3.1	4.8				
18	Patchouli alcohol C ₁₅ H ₂₆ O			9	9.6	11						

S. No	VOCs	P-1	P-2	P-3	P-4	P-5	S-1	S-2	S-3	T-1	T-2	T-3
19	(3,8,8-Trimethyl-1,2,3,4,5,6,7,8-octahydro-2-naphthalenyl)methyl acetate C ₁₆ H ₂₆ O ₂					3.7						
20	1,2,3,4,7,7a-Hexahydro-1,4,4,5-tetramethyl-6-acetyl-1,3a-ethano-3aH-indene C ₁₇ H ₂₆ O	9.1				4.8						
21	Dasycarpidan-1-methanol, acetate (ester) C ₂₀ H ₂₆ N ₂ O ₂							6	9.26			

Table 5

Fatty acid methyl esters present (peak area percentage) in aftershave lotion, face cream, hair dye and lipstick samples

S. No	Fatty acid methyl esters (FAME)	A-1	A-2	F-1	F-2	F-3	H-1	H-2	L-1	L-2
1	Palmitic acid methyl ester, C ₁₇ H ₃₄ O ₂			4.08		11.11				
2	13-Methylpentadecanoic acid methyl ester C ₁₇ H ₃₄ O ₂									
3	14-Methylpentadecanoic acid methyl ester C ₁₇ H ₃₄ O ₂	4.24							11.23	10
4	10-Octadecanoic acid methyl ester C ₁₉ H ₃₆ O ₂	12.15	10.99	14.29						
5	16-Octadecenoic acid methyl ester C ₁₉ H ₃₆ O ₂						44.44			
6	16-Methylheptadecanoic acid methyl ester C ₁₉ H ₃₈ O ₂					12.04				
7	Palmitic acid isobutyl ester C ₂₀ H ₄₀ O ₂								19.79	38.33

Table 6

Fatty acid methyl esters present (peak area percentage) in perfume, shampoo and talcum powder samples

S. No	Fatty acid methyl esters (FAME)	P-1	P-2	P-3	P-4	P-5	S-1	S-2	S-3	T-1	T-2	T-3
1	Palmitic acid methyl ester, C ₁₇ H ₃₄ O ₂							6.55	10.19			
2	13-Methylpentadecanoic acid methyl ester C ₁₇ H ₃₄ O ₂											
3	14-Methylpentadecanoic acid methyl ester C ₁₇ H ₃₄ O ₂											
4	10-Octadecanoic acid methyl ester C ₁₉ H ₃₆ O ₂		17.48				4.07	8.93	18.52			12.12
5	16-Octadecenoic acid methyl ester C ₁₉ H ₃₆ O ₂									9.13	8.86	
6	16-Methylheptadecanoic acid methyl ester C ₁₉ H ₃₈ O ₂		7.28							7.31		
7	Palmitic acid isobutyl ester C ₂₀ H ₄₀ O ₂											

In general, higher fatty acids can control the viscosity and dispersion properties of cosmetics. Such a higher fatty acid, 1-dodecanol is identified in the shampoo samples S-1, S-2 and S-3 with peak area percentage of 7.54, 7.14 and 10.19, respectively. It is a colorless insoluble solid alcohol used in the cosmetic products as emulsifier. It is an eye irritant and if unnoticed can affect the vision depending upon the idiosyncratic features (Veenstra et al. 2009). The exposure of 1-dodecanol may cause drowsiness and dizziness, loss of reflexes, vertigo, narcosis, reduced alertness and lack of coordination (Aleman et al. 2001; Bos et al. 1992). 1-Tetradecanol, a straight-chain saturated fatty alcohol, is found in the shampoo samples S-2 and S-3 with peak area percentage of 6.55 and 8.33, respectively. 1-Tetradecanol has been reported to affect the skin and lead to acute dermal disorders. It also produces health damage through abrasions, lesions, or wounds and at higher dosages, it can attack the cerebrospinal nerves. (Ashby and Tennant 1991). The aftershave lotion sample A-2 and talcum powder T-1 have 2,7-dimethyl-2,7-octanediol with a peak percentage of 2.41 and 8.22, respectively. Mild exposure to this compound leads to nausea. Overexposure of this compound may result in headache, dizziness, tiredness and unconsciousness (Steinemann 2009). Similarly, 3,7-dimethyl-6-octen-1-ol is observed in the samples P-1 and P-5 with a peak area percentage of 2.75 and 5.49, respectively, which may cause eye and skin irritation.

Under the alcohol category, benzyl alcohol is noticed in the samples P-3 and A-1. It is used in a wide variety of cosmetic formulations as a fragrance component, solvent, preservative viscosity-decreasing agent and pH

adjuster. Its acceptable daily intake is 5 mg/kg as per the recommendations of World Health Organization. Beyond that, it may cause drowsiness and dizziness in human beings. There are some evidences to suggest that the material can damage to lungs at extreme levels. Benzyl alcohol may affect the cardiovascular and respiratory systems through inhalation. On continuous exposure, it affects the central nervous system. In addition, it is also harmful to kidney and liver (Manjunatha et al. 2021). 2-(Dodecyloxy) ethanol is identified in all the shampoo samples (S-1, S-2 and S-3) with peak area percentage between 7.14 and 9.57. Its most adverse effect is irritation at the area of contact. It is also an environmental pollutant.

The volatile ester, 4-*tert*-butylcyclohexyl acetate identified in the samples F-1, F-2 and T-1 can cause eye and skin irritations. It may be harmful if it is absorbed through the skin. Inhalation of 4-*tert*-butylcyclohexyl acetate cause respiratory tract irritation (Belsito et al. 2008). 10-Methylundecanoic acid methyl ester is found in the samples P-4 and S-1. Inhalation of vapors may cause lack of co-ordination, loss of reflexes, drowsiness and dizziness. It can cause tear or conjunctival redness in the eye on direct contact (Sales-Campos et al. 2013). Diethyl phthalate is identified in P-1, P-3, P-5, A-1, A-2, L-1 and T-2 with a percentage peak area ranging between 5.35 and 12.82. It has been listed as an inevitable component of beauty products with a concentrations range < 0.1–28.6% (Api 2001). It has a tendency to linger in the mucous membranes than can lead to the damage of the lungs (Kamrin and Mayor, 1991). Inhalation of this vapor can cause the respiratory problems. Its exposure can cause headache, dizziness and nausea. Continuous exposure of diethyl phthalate can damage the central nervous system (Api 2001; Autian 1973).

Butylated hydroxy toluene (BHT), a derivative of phenol, is found in the samples F-1, L-2 and T-3. Used at a very low concentration to prevent the oxidation of other ingredients of a beauty product, it can penetrate the skin without causing any significant health hazards. It can cause respiratory irritation that leads to lung damage at higher concentrations (Lanigan and Yamarik 2002). It enters the body through inhalation, which can cause various health problems, such as anemia, convulsions, cyanosis, diarrhea, hypotension, inflammation of the lung, hyperventilation, nausea, and restlessness. In rare cases, overexposure may lead to respiratory failure and kidney damage (Nakagawa et al. 1984). Diethylene glycol monododecyl ether is observed in samples S-1, S-2 and S-3 with peak area percentage between 7.13 and 9.26. Triethylene glycol monododecyl ether is found in samples S-1 and S-2 with peak area percentage 7.94 and 7.14, respectively. These two compounds are relatively less toxic.

The VOC 1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[*g*]-2-benzopyran found in the samples L-1, L-2 and A-1 is not toxic to human, but hazardous to the aquatic environment. It is a synthetic musk with a clean sweet musky floral woody odor used in fragrances. Although its ability to enter into various systems of the human body, extensive research concluded that, it is safe for use in consumer products (Hutter et al. 2009).

D-Limonene, a monoterpene, is found in the samples P-3 P-4 and P-5 with a peak area percentage around 5. It is considered as an eye irritant and a skin irritant. The severity of continuous exposure is noted in humans in terms of irritation/scabies (Kim et al. 2003). Thiopropionamide is found in samples P-4 and P-5 with a peak area percentage of 3.64 and 3.66, respectively. Its target organs are respiratory system, gastrointestinal system and skin. It may cause eye irritation. Thiopropionamide is harmful if it is absorbed through the skin. The other major FAMES have been classified as irritants. On evaporation, the aerosols may cause fatigue and vomiting sensation (Wertz and Downing 1990).

Overall, the present study has found the presence of many toxic materials as the prominent ingredients in the cosmetic products manufactured and marketed in South India. Although, most of the VOCs are found in other common personal cleanliness products, consumers should be warned about their use considering the severe effects of certain compounds. Moreover, the study urges the manufacturers to inform the public of the environmental impact of all the ingredients of the products irrespective of their concentration used.

Conclusion

Cosmetic products used on a daily basis have been reported to contain a variety of volatile organic compounds. The GC-MS analysis of samples of selected personal care products manufactured in South India have been shown to contain certain highly toxic VOCs in alarming levels. Diethyl phthalate, which is a potential environmental pollutant and toxicant causing mild to severe ailments in human beings, was found in majority of the samples. Apart from this, the presence of many hazardous VOCs and an array of fatty acid methyl esters has been established by this preliminary study. Thus, the study suggests the proper display of the type of ingredients used in the products with specific warning notes. In the absence of such messages, consumers would be masked from the harmful nature of chemical used in the formulation of cosmetic products. Through this research, appropriate regulatory bodies are requested to curb any illegal practices in the manufacture and marketing of these products. A detailed clinical report on the side effects of these harmful compounds on the health of consumers would be quite useful. Another option is to find environmentally friendly viable alternatives that can substitute the existing toxic ingredients as a remedial measure to reduce the detrimental effects on human health.

Declarations

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Dr. A. Ramasubbu: (Corresponding author)- Design of the work and approved the version to be published

R. Mayildurai: Execution of the research work assigned.

A. Srinivasan: The research work was co-executed with appropriate research tools.

Dr. T. Maruthavanan: interpretation of results and revised it critically for important intellectual content

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