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Thermogravimetric and Thermogravimetry - Mass Spectrometric study of Selected Incense Sticks manufactured in India

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(Dedicated to Prof.U.Murali Krishna,
Former Professor of Analytical Chemistry, Andhra University On his 80th birthday celebrations)

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ABSTRACT

Thermal behaviour of ten Indian made Incense Sticks extensively used by public were studied using thermogravimetry (TG) in the temperature range 30– 850°C in nitrogen and zero air atmospheres and emitted gases were characterized using thermogravimetry – mass spectrometry (TG-MS). The thermal behaviour of all types of incense sticks showed five significant mass loss stages in nitrogen and air atmospheres. The first three stages indicate evaporation of gaseous materials and the rest may be due to thermal decomposition processes. Among these incense sticks, only two brands showed a slight deviation and other eight brands exhibited similar thermal behaviour. From the TG-MS analysis, the nature of gases emitted from different brands of selected incense sticks are similar with same functional groups. The extent of gaseous emissions is different for different brands, but we could not quantify them.

Keywords: Thermogravimetry, Thermogravimetry- Mass Spectrometry, Incense Stick, Decomposition and Emitted gases.

INTRODUCTION

Through out the world, incense sticks are extensively used in holy places in countries like China, Taiwan, Malaysia, Indonesia, India etc. and to some extent in other countries. Incense sticks are generally known as Agarbathi in India. Many studies revealed that exposure to smoke emitted from incense sticks is adverse to human health.

The exact composition of incense sticks is not reported by any manufacturer. But, in general, a typical composition of incense stick is about 21% of herbal and wood powder, 35% of fragrance materials, 11% of adhesive materials, and 33% of bamboo stick [1].This diverse composition of incense sticks results in the release of gases of different nature in the indoor environment during their burning process. People are inhaling and are in contact with those emitted gases. In general women, children, and elderly people stay at home for long time and are exposed to those gases emitted from incense sticks.

The extracts of incense smoke are reported to be mutagenic in the Ames Salmonella and Salmonella typhuimurium tests [2-4]. Association of childhood brain tumors [5] and risk of childhood leukemia [6] in

children are reported that are exposed to smoke from incense stick burning in their home. Friborg et al [7] reported association of incense use with increased risk of respiratory carcinomas in the entire respiratory tract. Incense burning was associated with coughing symptoms in children [8] while others exposed to the emissions showed significant chronic cough and acute irritative symptoms [9]. As adverse health effects of incense smoke are related to the composition of smoke, researchers [10-19] tried to characterize the gases in the smoke from incense sticks in terms of emissions of volatile organic compounds (VOCs) [10,11]; in terms of emissions of polyaromatic hydrocarbons (PAHs) [12-14]; in terms of emission of carbonyls and reactive oxygen compounds [15-18]; and in relation to their metallic content [19].
There is no report on the thermal behaviour of Indian made incense sticks and the composition of emitted gases. We report here the TG studies of ten different most widely used brands of incense sticks in India and the composition of incense smoke as a function of temperature using TG-MS technique.

MATERIALS AND METHODS

Selection: Ten brands of incense sticks, most commonly used by public in India were purchased from the local markets. In Table 1, details of selected incense stick samples are presented.

Table 1: Selected Incense sticks for thermal analysis

Sample No.	Brand Name
1	Mangaldeep Bouquet
2	Dasara Yoga
3	Swarna Champa
4	Swarna Lavender
5	Shivling Champa
6	Rhythm Amber
7	Chola
8	Lucky Kewda
9	Ambica Durbar Bathi
10	Paradise

Thermogravimetric analysis: A Mettler Toledo TGA/SDTA851^{e} model thermobalance with star^{e} software was used for thermogravimetric analysis. A method with a heating rate of 3°C per minute in the temperature range of 30-850°C was used. The thermograms were obtained both in Nitrogen and air atmospheres. Nitrogen gas was used as a purging and protective gas with flow rates of 80mLmin^{-1} and 40mLmin^{-1}, respectively. Zero air was used as reactive gas with a flow rate of 60mLmin^{-1}. About 30 mg of sample was weighed in a 150 µL alumina crucible and introduced into the thermobalance. The weight loss of the sample as a function of temperature was recorded and analysed.

Thermogravimetric-mass spectroscopy (TG-MS) analysis: A hyphenated PerkinElmer TG-MS was used for the identification of the evolved gases from incense burning. About 8 mg of the sample in a platinum crucible was used. The temperature programme for TG-MS was from 20°C to 600°C at a heating rate of l0^{O}C/min. Helium gas was used at a rate of 50 ml/min. The mass spectrometer detector was operated in an electron impact mode with 70eV, 200°C source temperature and 10-500amu scan range. The transfer line temperature was set at 200°C.

RESULTS AND DISCUSSION

TG analysis: Fig.1A and Fig.1B present the thermograms of typical incense stick,Chola; Sample No:7 in nitrogen and zero air atmospheres. From the results,the thermal behavior of incense sticks in nitrogen atmosphere can be divided into five stages as shown in Table 2. The first stage of mass loss within the temperature ranges from 30 to 110°C, the second stage is 110- 210°C, the third stage starts from 210-410°C, the fourth stage is 410-550°C, and the last stage is from 550 -750°C. It can be seen that the mass loss in stage four is comparable to the mass loss in the first stage but the change in the mass as a function of time as well as temperature is slow in the former leading to a very weak trough as compared to the other stages.

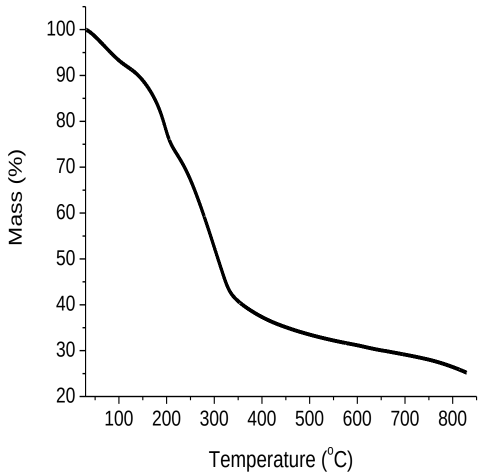

Fig 1A: Thermogram of the sample Chola (7) in Nitrogen Atmosphere

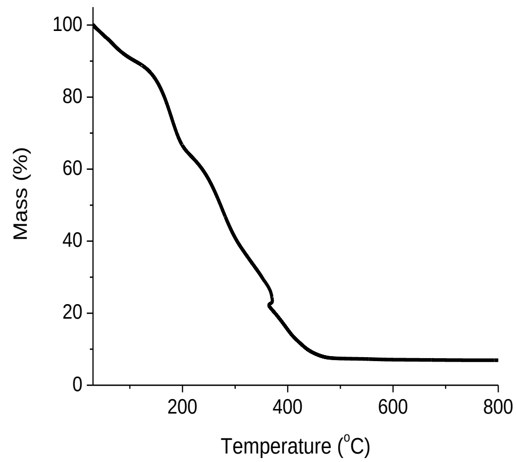

Fig 1B: Thermogram of the sample Chola (7) in Air Atmosphere

As it is shown in Table 2, the order of mass loss in most samples is 2nd stage > 3rd stage > 5th stage > 1st stage ~ 4th stage. Some deviations are observed from this order as in the case of sample 10 which exhibited lower mass loss in the 2nd stage. But a significant change is observed in samples 7 and 9, whereas Sample 7 showed maximum mass loss in the third stage. Beyond 410°C, the loss in mass is not considerable. In the case of sample 9, the second stage is split into two steps of mass loss and the third stage is the one that showed maximum mass loss (~ 47%).

Table.2: The five stages and associated percent mass losses of incense sticks in nitrogen atmosphere

Sample No	Mass loss(in Percentage)				
	1st stage (upto 110°C)	**2nd stage (110-210°C)**	**3rd stage (210-410°C)**	**4th stage (410-550°C)**	**5th stage (550-750°C)**
1	7	20	18	7	10
2	5	20	15	7	10
3	4	20	16	6	11
4	6	22	18	7	11
5	5	27	16	4	8
6	5	25	13	4	5
7	8	19	39	4	3
8	6	23	18	4	8
9	4	17	47	6	5
10	6	8	21	7	13

Similarly, the thermal behavior of incense sticks in air are similar in most of the samples, except those of 7 and 9, which exhibited five stages of thermal and oxidative decompositions. Table 3 presents the percent mass loss in the possible five stages of incense burning process. The first step is most likely the

evaporation of highly volatile components up to 100°C, the second stage is from 100 - 200°C,and other stages are third stage :200-350°C, fourth stage : 350-550°C, and fifth stage :550 to 670°C.
The first three stages both in nitrogen and air atmospheres are similar, except with minor variations, signifying evaporation and thermal decomposition processes dominating in these stages. Despite the slow decomposition rate observed in the fourth stage in nitrogen atmosphere (410-550°C), the fourth stage in air atmosphere (350-550°C) showed a significant mass loss and rate of mass loss indicating that the oxidative decomposition is significant. The emission of gases continued up to 670°C in the oxidative environment, where as it is up to 750^{0}C in the nitrogen atmosphere. Generally, the mass loss in the stage four is high compared to the other stages.

Table.3: The five stages and associated percent mass losses of incense sticks in air atmosphere

Sample No	**Mass loss(in Percentage)**				
	1st stage (up to 100°C)	**2nd stage (100-200°C)**	**3rd stage (200-350°C)**	**4th stage (350-550°C)**	**5th stage (550-670°C)**
1	7	15	21	28	3
2	6	17	17	28	4
3	6	15	19	28	5
4	6	17	21	26	3
5	6	20	19	25	4
6	6	14	17	32	3
7	10	25	35 (200-330°C)	14 (330-370°C)	14 (370-560°C)
8	7	5	25	30	4
9	5	40(100-330°C)	-	49 (330-500°C)	-
10	8	3	23	35	6

Samples 7 and 9 showed deviations from the pattern compared to the other samples. In air environment, the fourth stage in sample 7 is divided into two steps. A high rate in mass loss is observed in the temperature range 330-370°C followed by another step in the range 370-560°C. The mass loss beyond 560°C is not significant. Similarly, in air atmosphere, sample 9 showed three stages, as shown in Table 3. There is no mass loss beyond 500°C.

TG-MS analysis: Table 4 presents the nature of emitted gases from incense sticks as analyzed by TG - MS technique. Despite the drawback of TG - MS for analyzing complex gas mixtures, it can lead to some extent; characterize the gases released as a function of temperature as the technique is a real time analysis. As it can be seen from Table 4, each burning stage is accompanied by release of specific gases depending on the formulation of the incense sticks. In 1st stage water and 2,6- Dimethyl- 7-octen- 2-ol are predominant volatiles. Most of incense sticks emitted Phenyl ethanol in the 2nd stage and Glycidol in the 3rd stage. The occurrence of Carbon dioxide and Benzene in 4th stage may indicate the burning process. In this study, despite the expected numerous compounds, only few gases are identified from incense stick samples as TG - MS technique has no resolution capabilities. Fig.2 shows the total ion mass chromatogram. Yang et al [10] identified and quantified 17 volatile organic compounds while Eggert et al [11] could identify 96 gases but quantified only 14 gases during incense stick burning. Both of them [10, 11] reported the presence of 2,6- Dimethyl- 7-octen- 2-ol and benzene.

Table.4: Nature of gases emitted during incense burning

Sample No	1st stage (upto 110°C)	2nd stage (110-210°C)	3rd stage (210-410°C)	4th stage (410-550°C)
1	Water 2,6- Dimethyl- 7-octen- 2-ol 2-methyl- 6-methylene	Anthracene	Acetic acid Glycidol	Carbondioxide
2	Water 2,6- Dimethyl- 7-octen- 2-ol	Phenyl ethanol	Glycidol	Carbondioxide Benzene
3	Water 2,6- Dimethyl- 7-octen- 2-ol	Phenyl ethanol	Glycidol Diethylphthalate	Carbondioxide
4	Water, 2,6- Dimethyl- 7-octen- 2-ol	Aromadendrene2	Glycidol	Carbondioxide Benzene
5	Water 2,6- Dimethyl- 7-octen- 2-ol		Glycidol	Carbondioxide Benzene
6	Water 2,6- Dimethyl- 7-octen- 2-ol	Phenyl ethanol	Glycidol	Carbondioxide Benzene
7	Water 2,6- Dimethyl- 7-octen- 2-ol	1-butanol-4-(ethenyloxy)	Glycidol	Carbondioxide, Benzene
8	Water,Cyclohexanol, 5- methyl- 2-(1 methylethenyl)	Phenyl ethanol	Glycidol	Carbondioxide
9	Water	Phenyl ethanol	Acetic acid Glycidol	Carbondioxide 2-heptanal
10	Water 2,6- Dimethyl- 7-octen- 2-ol	2-hydroxy valerophenone	Glycidol	Carbondioxide Phenethylamine

APPLICATIONS

The results are useful to find the nature of gases emitted from different brands of selected incense sticks.

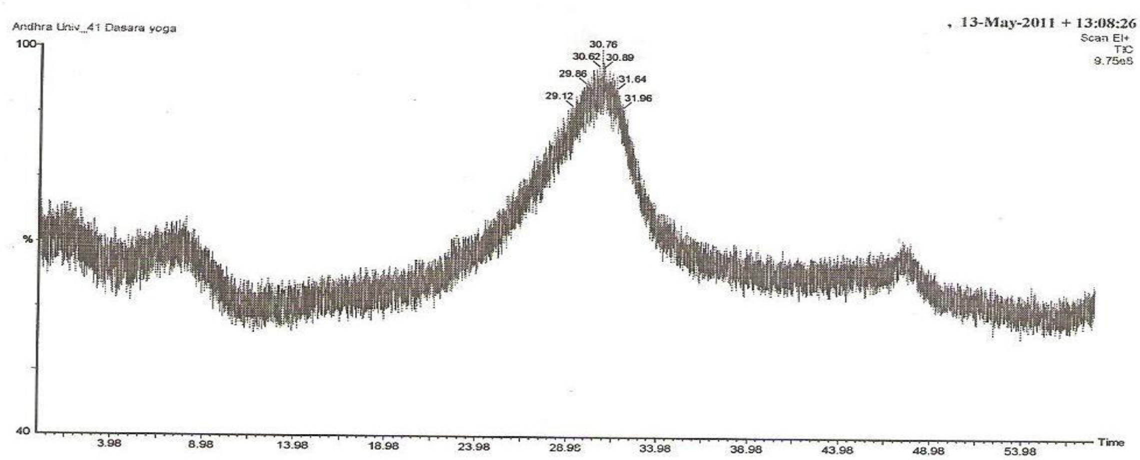

Fig. 2. Typical Total ion chromatogram of sample (2) from TG-MS analysis

CONCLUSIONS

The thermal behaviour of 10 incense sticks have been studied and among them eight incense sticks showed similar behavior and the other two showed different behavior. The thermal behavior of incense sticks can be divided in to five stages both in nitrogen and air atmospheres. In inert atmosphere the four stages are quite significant while the five stage classification is found to be precise for burning incense sticks in air. The fourth stage in the temperature range 350-550°C is found to be the region where oxidative degradation is dominating in all brands of incense sticks.

The emitted gases are identified in TG – MS in first stage, up to 110°C two compounds water and 2,6-Dimethyl- 7-octen- 2-ol and in last stage, 410-550°C Carbon dioxide and Benzene are identified. Benzene is known to cause health risk where as 2,6- Dimethyl- 7-octen- 2-ol is reported to have no health risks[11]

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