A case study on the low probability extinction of smouldering incense sticks

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Abstract

The problem of a low probability extinction of smouldering incense sticks was posed by a leading agarbatti industry and it was intended to determine the cause of this problem. This led to a series of investigations that covered the measurement of smouldering rate and surface temperature of a number of compositions, some of which were supplied by the industry, some bought from outside and some prepared at the laboratory. The study dismissed the initial thoughts on the possible causes – the presence of phosphorous, potassium, sodium related compounds ash fraction of the incense material and finally related to a combination of chemicals coming from termite infested wood that would have been the part of the supply chain of the industry.

Introduction

The incense stick industry is prevalent in religiously dominated eastern countries – India and south east Asia more than others. The major composition of incense stick is almost constant for all types of incense sticks irrespective of different industries and brands. Typically, an incense stick comprises finely ground powder of wood, charcoal and adhesives and pasting the material on a bamboo stick of about 1.5 mm size [1,2]. It is then impregnated with fragrance material. The role of fragrance material is that is actually an intellectual property of the specific industry and is not of any relevance to the present study as it constitutes a tiny fraction of the composition. This work studies the problem of a low probability extinction of smouldering incense sticks posed by a leading agarbatti manufacturing industry. The industry facing the problem provided the composition of what constitutes "good" sample and "bad" sample. The industry also indicated that the rejection of "bad" samples was to the extent of 0.1 to 0.2 % and this did not appear significant. But then, the industry pointed out that the incense sticks when used on auspicious occasions should function normally and if even one of them extinguishes, the matter is of deep concern due to the impression of this being indicative of something being inauspicious. It was in this backdrop investigations were undertaken.

Experimental studies

A thorough survey through the literature also indicated the works conducted so far were related to either ill effects of incense sticks, air pollution, product developments or understanding composition and smouldering process of incense sticks [1-7] rather than the problem of smouldering as posed by the industry as in the present case. In order to understand the behaviour, samples of the ingredients and several batches of incense sticks were sought. Also, samples from other brands were bought from the market. They were mounted on a vertical stand having holes on its flat surface in a 10 x 10 square matrix. Each hole can hold one stick at a time. Smouldering of the sticks was initiated on this stand by igniting with the help of safety matches. Simultaneously, the red hot glowing region was examined using a thermal camera (make: FLIR – T62101). In addition, chemical investigations using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy and Energy Dispersive X-ray diffraction (SEM/EDX) , Thermogravimetry DT/TGA and X- Ray Diffraction (XRD) were also conducted in a progressive approach towards unravelling the issue. There were many misses, but one clear hit! These are discussed below.

Results and Discussions

Burn Tests

All the data on the samples studies are listed in Table 1. The various terms used are explained in the table. Several experiments were done by igniting the samples to get a comparison on various parameters influencing the smouldering rate of the candidate problem sample (CB1 in the Table 1) with that of samples bought from the market. These generally showed that most of them were smouldering at rates of 2 to 3.6 mm/min. The data on the first two samples shows that the first one is smouldering well, but the second one extinguishes after some time. The data from thermal camera is set out in Figure 1.

Table 1 The data on the incense sticks (M-1, etc = Other market samples, CGo -1 = Good sample from reference industry; CB – 1 = Bad sample from reference industry); Cgar -1, 2,3 = Gaur gum related samples made in the laboratory; C1 to C6 = different compositions specially made to determine effect of charcoal; S/T = Sand or termite infested wood powder

Sample	Powders													
	Wood %	Charcoal %	Adhesive %	Gaur gum %	S / T	L cm	Dia mm	f_w %	m_o g	ρ kg/m³	Ash %	$\dot{r}$ mm/min	$\rho\dot{r}$g/m²s	T_s, °C
CGo-1	40	45	15	0	0	20.5	3.08	8	1.63	1067	15.1	3.6	64.3	640
CB - 1	40	45	15	0	0	18.7	3.26	8	1.38	880	20.0	Ext	-	530
M-1	NA	NA	NA	NA	NA	17.8	3.16	8	1.39	998	7.9	2.6	43.4	630
M-2	NA	NA	NA	NA	NA	22.8	3.12	8	1.94	1111	14.7	3.5	65.3	610
M-3	NA	NA	NA	NA	NA	20.1	3.08	8	1.67	1112	14.1	2.5	46.3	640
***M*-4**	NA	NA	NA	NA	NA	18	2.82	8	1.01	899	6.9	2.9	42.8	615
M-5	NA	NA	NA	NA	NA	18	2.96	8	1.01	813	20.9	3.3	45.2	620
M-6	NA	NA	NA	NA	NA	17.5	3.18	8	1.22	876	14.8	2.9	41.7	630
Cgar-1	33	50	8	9	0	4.14	8.63	3	1.62	668	18.8	2.2	24.0	610
Cgar-2	33	50	8	9	0	4.11	8.56	8	1.79	755	17.1	1.6	19.6	560
Cgar-3	33	50	8	9	0	4.14	8.46	15	1.72	740	17.7	1.5	18.5	600
C1	33	37.5	12.5	0	**17S**	3.72	8.73	5	1.69	760	35	1.91	24.2	630
C2	27.5	31	10.5	0	**31S**	3.78	8.76	5	1.92	844	41.5	1.83	25.8	600
C3	28.5	32	10.7	0	**29 T**	2.38	8.5	5	1.08	800	-	Ext	-	500
C4	85	0	15	0	0	3.93	8.78	5	1.29	542	12.5	2.3	20.5	620
C5	0	85	15	0	0	3.67	8.85	6	1.74	770	23	2.2	28.4	610
C6	40	45	15	0	0	3.97	8.79	5	1.59	662	20	2.5	27.8	640
C7	45	40	15	0	0	3.51	8.71	6	1.37	657	19	2.2	24.4	640
C8	65	20	15	0	0	3.82	8.74	6	1.35	591	14	1.9	18.9	605
C9	20	65	15	0	0	3.63	8.71	7	1.53	706	23	2.6	30.4	635

Surface temperature during smouldering was observed using FLIR T62101 model Thermal imaging camera. The temperature remains steady around 630 °C for the good sticks and in the case of bad sticks it climbs to around 450 to 500 °C and drops off continuously. The initial suspicion was with regard to ash fraction. It was thought that with one of the ingredients – wood powder or the adhesive powder, there could have been an inadvertent mix of inorganic material – like mud or sand

and this may have resulted in the extinction. Several sticks of commercial nature were procured and tested (M1 to M6). One of these, M5, showed an ash fraction of 20.9 % and good steady smouldering rate of 3.3mm/min with a steady surface temperature of 620 °C. This put forth the question of ash fraction affecting the smouldering to question. An examination of literature [8] showed that phosphorous compounds are used as inhibitors for smouldering or combustion. For instance, Lowden and Hull (2013)[8] state that "phosphorus-based compounds are some of the best-known fire-retardant treatments for timber. They are considered to work mainly in the condensed-phase, by promoting char formation and depriving the gas-phase of further volatile decomposition products". Their method of working is described as that in the condensed-phase, the thermal decomposition of inorganic phosphorus-based compounds forms phosphoric acid, which condenses to form pyrophosphate and water.The water released dilutes the oxidising gas-phase and both phosphoric acid and pyrophosphate promote the dehydration of wood's terminal alcohols, cross linking the cellulose structure, promoting wood's natural ability to char. While the retarding property was thus a well-established feature, what was unclear was whether specific woods grown in solid with extensively fertilized soils would have so much of phosphorous as to cause natural fire retardancy.

During the study several discussions took place with the technical team of the industry and at one time they indicated samples prepared from gaur gum as the adhesive has problems of extinction (around 9 %). For examining this question, samples were prepared with gaur gum (Cgar 1 to 3 in Table 1) and these smouldered very well and it was not possible to attribute the associated problems to gaur gum.

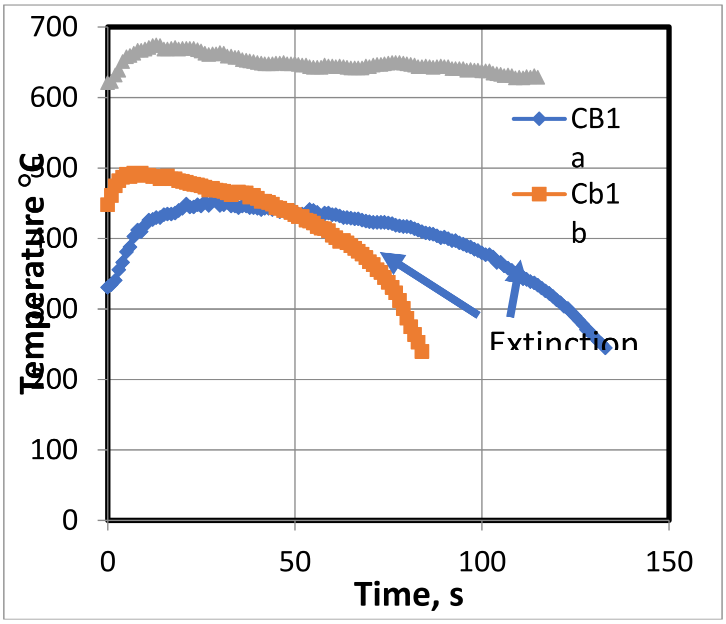


Figure 1: Temperature Vs time data for good and bad sticks

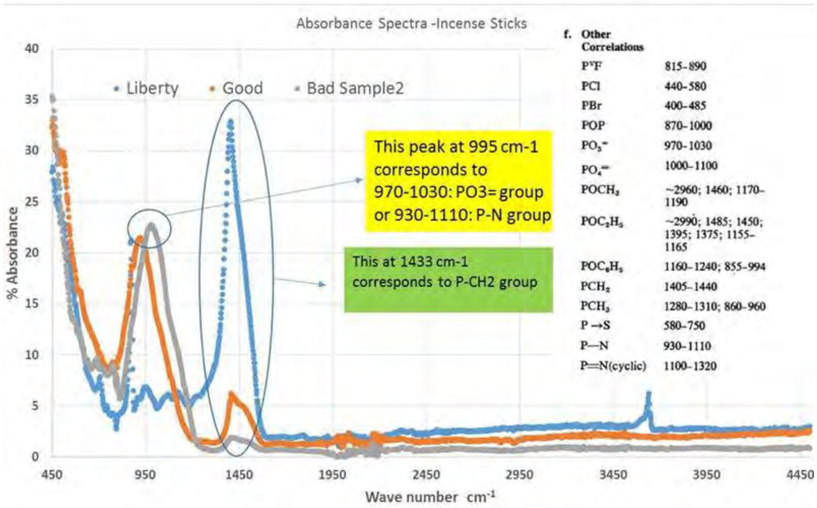
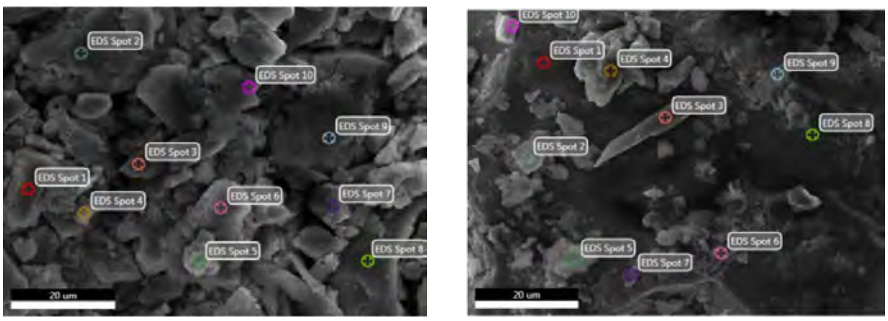


Figure 2: Absorbance FTIR spectra of the incense sticks. (above figure to be replaced)

FTIR and SEM/EDX studies

The work of Ovington and Madgwick (1958)[9] showed that the contents of potassium, sodium and phosphorous are about between 0.03 to 0.1 % of the biomass and so, the naturally ingrained compounds would most likely not responsible for the observed extinction. Seeking confirmation of this thought, the samples were subject to Fourier Transformed Infrared spectral analysis. Figure 2 shows the spectra. Both the good and the bad samples have similar peaks and it is unlikely that phosphorous related compounds make the difference. To examine whether the bad samples has any other element that could cause the problem, the samples were subject to SEM/EDX study. Figure 3 shows the SEM images of good and bad samples in which 10 spots were picked and averaged for EDX analysis. The results are set out Table 2. Using this data possible combinations of oxidized compounds that could be present naturally in wood is arrived (Table 3). These showed that Si in all likelihood as SiO_2 could be suspected as a candidate for the observed extinction. This was also because sand could get mixed with biomass when it is being pulverized in a not-so-clean an environment and sand contributing silica was thought responsible.



Good Bad

Figure 3: SEM images of good and bad samples with EDX spots

Table 2: Elemental composition as indicated by SEM/EDX analysis of good and bad samples

Good

Element	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6	Spot 7	Spot 8	Spot 9	Spot 10	Avg
B	4.3	5.6	0.4	5.7	0.3	0.2	5.5	5.3	5.9	5.4	**3.9**
C	61.7	63.6	87.7	74.7	40.2	58.4	65.3	88.3	76.9	71.5	**68.8**
N	0.0	0.0	2.3	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.3
o	21.1	23.2	7.9	14.0	40.5	21.2	23.7	5.0	11.9	14.6	**18.3**
Mg	1.4	1.2	0.1	0.3	0.9	0.4	0.2	0.0	0.6	0.4	0.5
Al	0.5	0.3	0.1	0.2	0.6	0.9	0.2	0.0	0.3	0.5	0.4
Si	0.9	0.4	0.1	0.6	0.9	1.6	0.5	0.0	0.4	0.6	0.6
P	2.9	1.5	0.3	1.8	2.4	5.6	1.8	0.9	1.4	1.9	**2.0**
Cl	0.4	0.2	0.3	0.3	0.2	0.6	0.1	0.0	0.3	0.5	0.3
K	2.9	2.0	0.1	0.5	0.6	2.6	1.4	0.3	0.8	0.5	1.2
Ca	3.8	2.2	0.7	1.9	13.6	8.5	1.1	0.1	1.6	4.0	**3.7**
	100.0	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**

Bad

Element	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6	Spot 7	Spot 8	Spot 9	Spot 10	Avg
B	0.1	4.9	8.0	5.4	5.6	5.2	5.2	0.1	0.1	0.8	**3.5**
C	4.6	39.3	78.0	71.5	43.5	54.0	55.9	4.2	24.1	47.5	**42.3**
N	0.6	2.6	0.0	0.0	4.3	0.2	2.0	0.3	0.0	0.0	1.0
o	47.0	36.6	9.9	21.1	34.7	19.9	21.3	46.9	31.8	28.1	**29.7**
Mg	0.3	1.1	0.0	0.0	0.9	0.1	0.1	0.2	0.2	0.0	0.3
Al	0.6	2.5	0.1	0.0	1.4	0.4	0.6	0.4	2.2	0.1	0.8
Si	45.9	8.0	2.1	0.3	6.7	19.3	14.0	47.3	34.8	21.7	**20.0**
P	0.6	1.0	0.8	1.0	0.9	0.5	0.5	0.5	1.7	1.2	0.9
Cl	0.1	0.2	0.0	0.0	0.1	0.1	0.0	0.1	0.3	0.0	0.1
K	0.2	1.6	0.1	0.5	0.8	0.1	0.2	0.0	1.3	0.5	0.5
Ca	0.1	2.2	0.9	0.3	1.3	0.2	0.4	0.1	3.5	0.1	0.9
	100.0	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**

Summary

Element	Atomic Mass	% Inorganics Good	Bad
B	10.8	3.9	3.5
C	12.0	68.8	42.3
N	14.0	0.3	1.0
o	16.0	18.3	29.7
Mg	24.3	0.5	0.3
Al	26.9	0.4	0.8
Si	28.1	0.6	20.0
P	31.0	2.0	0.9
Cl	35.5	0.3	0.1
K	39.1	1.2	0.5
Ca	40.0	3.7	0.9
		100.0	**100.0**

Table 3: Converting the element composition data to probable inorganic material composition

Probable Compounds	Good	Bad	Retarding Potential
B_2O_3	12.42	11.36	Yes
C	67.71	41.99	No
KCl	0.61	0.17	Yes
MgO	0.91	0.46	Yes
Al_2O_3	1.02	2.27	Yes
SiO_2	**1.27**	**34.23**	Yes
P_2O_5	4.69	1.99	Yes
$CaCO_3$	9.37	2.29	Yes
Others*	2.01	5.24	-
	100.00	**100.00**	

*** Nitrogen compounds are considered here**

Two experiments were made by adding significant amount of fine pulverised sand into the composition. These are set out in Table 1 as C1 and C2. The ash content was taken to as high a value as 41.5 %. The sticks seemed to smoulder at lower rates of > 1.8 mm/min with surface temperature in the range of 600 °C+. This ruled out silica alone as a possible candidate and shifted the thought process towards other forms of silica or combinations of silica and silicates entering the product through sources like clay/mud, etc. This led to the choice of termite infestation of wood products as termite mounds are alumina phyllo silicates, complex compounds consisting of Silica as one of the major component along with poly hydroxyl groups of Magnesium, Aluminium and Sesquioxides of Iron and Manganese. In desperation (!), the termite ridden wood was considered a candidate for the problem. Specifically, composition C3 was produced to examine this speculation. It showed that the stick extinguished with surface temperature dropping to below 500 °C. The termite ridden wood used in this experiment was sourced from within the laboratory.

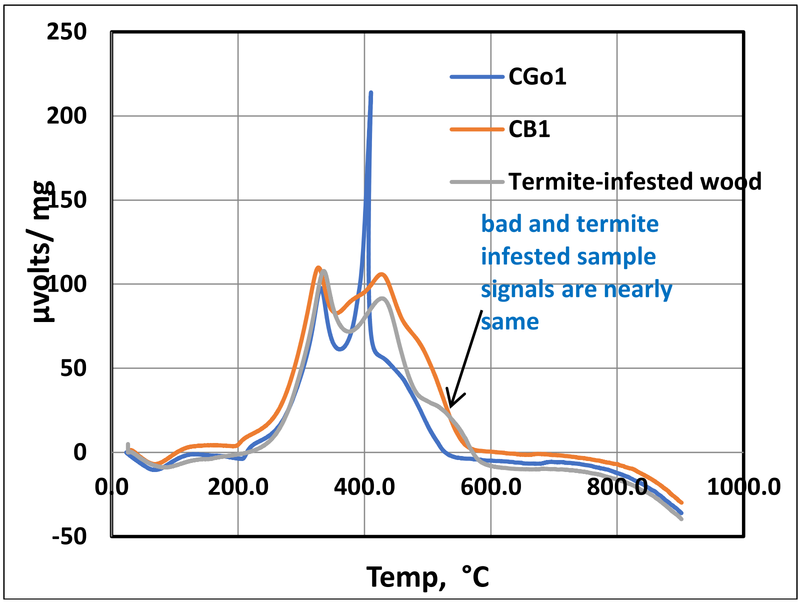


Figure 4: The DTA record of good, bad and termite infested samples

DT/TGA studies

It was necessary to examine if this feature is also a possibility in the reference industry case. To examine that DTA analysis was mounted for both the samples – CB1 and C3. The DTA comparison is set out in Fig. 3. It is very clear that the DTA records of the bad sample virtually coincide with that termite infested material – most probably from wood or adhesive material.

It is appropriate to infer that the composition of other termite muds may also be similar and one would need to determine the compound that gives endothermicity around 550 to 580°C and antigorite provides the possibility of high temperature decomposition, particularly because it contains 4 OH radicals that may break down at high temperature. Reviewing of literature showed the presence of serpentines like antigorite that are rich in hydroxyl groups in its complex structure and these may break down at high temperature. Hrsak et al (2008) have described the thermophysical properties of serpentinite that contains antigorite. They specifically have shown that the decomposition of antigorite occurs at around 660°C. Zulumyan et al (2017) have described the procedures used for analysis of the termite mud and Ferrand (2019) has brought out that the decomposition of antigorite occurs in steps - first, a slow de-hydroxylation process that breaks low-energy OH bonds and then, high-energy OH bonds are broken.

X-Ray Diffraction Studies

In order to understand the nature and composition of termite mud soils local to Karnataka (INDIA), samples of termite mud were collected from within the campus (fig!) and the samples were subjected to thermal studies and XRD studies. XRD studies were also conducted for the agarbatti samples received from the manufacturer.

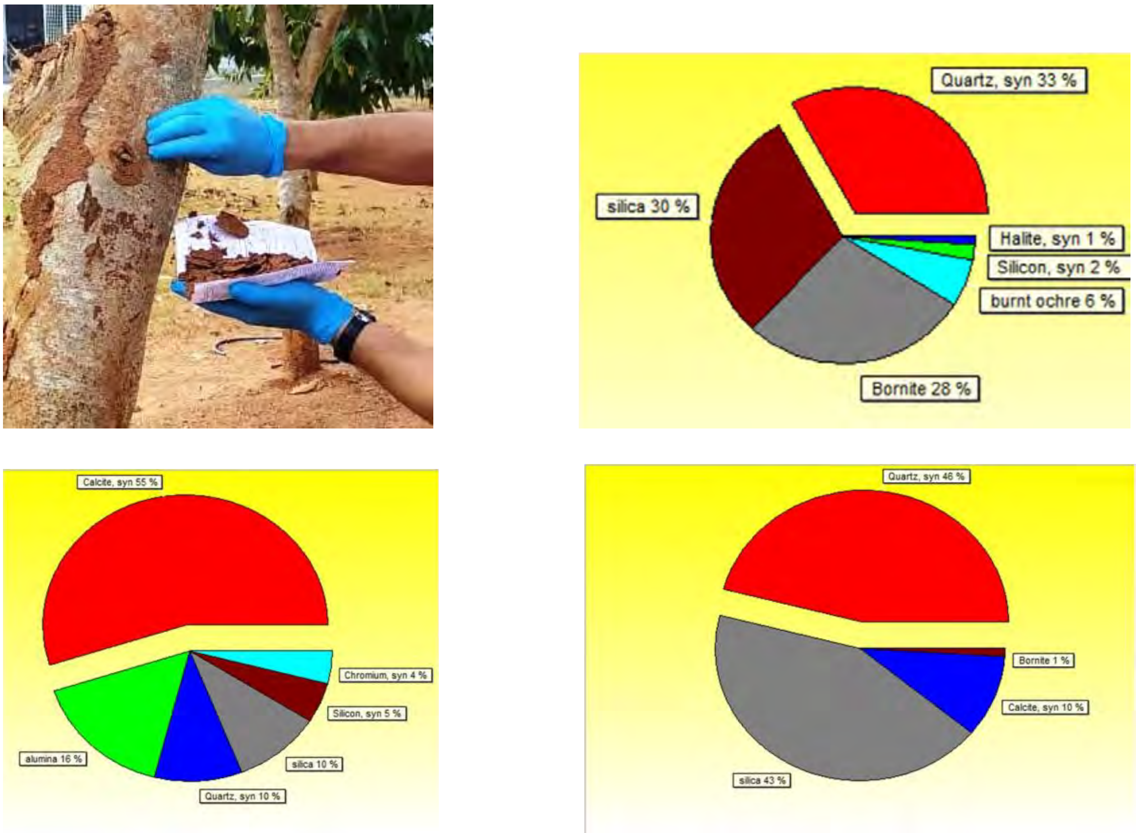


Fig 5. Termite mud collection and XRD interpretation of Good, Bad and Termite mud sample

Table 4: Calculated Mineralogical Composition

Composition	Termite mud	Bad sample	Good Sample
Silica (quartz), SiO_2	33 %	46%	10%
Silica (sand), SiO_2	30 %	43%	10%
Calcite	-	10%	55%
Alumina	-	-	16%
Bornite	28%	1%	-
Others (Si,Cr)	9%	-	9%
Total	100%	100%	100%

Results of XRD on the inerts are quantified through Rietveld refinement method using Panalytical software indicate the major presence of SiO_2 in the form of α-quartz. Nobel Laureate Sir CV Raman etal (1940) discuss about the abrupt changes to quartz at 575 °C, where α-quartz transforms to β-quartz. While the former has trigonal structure and the latter has hexagonal structure. This abrupt change could also be the reason for absorption of energy resulting in endothermicity at temperature 550 to 580 °C. Since XRD studies were conducted on green samples of the Agarbatti and in order to ascertain the results obtained above additional XRD studies are carried on the ash samples of good, bad and termite samples. These results are presented in the Table (samples to be analysed – slot awaited at CNMS)

Suggestions for improvement and cost reduction

The incense stick manufacturers use charcoal in fairly large proportions. If we recognize that every kg of charcoal uses traditional retorts or even in open fields and so, consumes 4 to 6 kg of wood in the process. In order to determine the importance of charcoal in smouldering process, several compositions were made with different proportions of wood: charcoal as in compositions C4 to C9. These results show that even though there are variations in the smouldering rate as a function of composition, there seems to be no specific order and the surface temperatures have been in the range of 600 °C+ which is a clear indication that smouldering is not enhanced in any serious way by charcoal. Therefore there is a distinct possibility of cost optimization with the proper choice of materials.

Concluding remarks

This work was undertaken to seek a possible cause for the observed extinction of some incense sticks and to examine if there is a simple and clear cause for it. It appears that termite infested wood related powder should be clearly avoided. Also charcoal that may cost much more can actually be eliminated or at least reduced in its fraction to optimize on the costs. As a part of scientific study, it is conceived that termite poop related composition be investigated beyond SEM/EDX technique.

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