PROJECT REPORT

ON

EVALUATION OF SUITABILITY OF SUGARCANE WAX FOR APPLICATION IN COSMETICS (Study I)

SUBMITTED TO

M/s GODAVARI BIOREFINERIES

MUMBAI

SUBMITTED BY

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1.0	**Title of the Project**	Evaluation of suitability of sugarcane wax for application in cosmetics
2.0	**Duration**	06 months (15-05-2015 to 15-11-2015)
3.0	**Sponsor**	M/s Godavari Biorefineries, Mumbai
4.0	**Organization**	Shriram Institute for Industrial Research 19, University Road, Delhi 110007
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1.0 Background

Sugarcane wax is attracting attention as a substitute of the natural waxes. Sugarcane wax is found on the cane bark surface as a whitish to dark yellow waxy substance. Its major function is the protection of bark surface. The consumption of natural waxes like carnauba and candelila waxes has increased in various application areas such as lubrication of machinery, polishing of metallic surfaces, manufacture of cosmetics, food ingredient, etc. As a result, their demand has also increased.

It is extracted from the press mud or filter cake mud, obtained after sugarcane juice clarification, by solvent extraction technique. Sugarcane wax is complex and variable mixture of long chain alkenes, hydrocarbons, fatty acids, ketones, aldehydes, alcohols, esters, and steroids. Sugarcane wax is indigestible and harmless to health. In its refined form it has a light yellowish colour. Due to the high melting point of 75 to 80 °C sugarcane wax remains stable even on exposure to direct sunlight.

The potential areas of sugarcane wax include pharmaceutical preparations, confectionary items, processed food, carbon papers, carbon less carbon papers, electrical insulations of cables and wires, paints & varnishes, crayons, coloured pencils and cosmetics.

M/s Godavari Biorefineries, Mumbai is commercially producing sugarcane wax under the trade name of “nautowax” and interested in its application research in cosmetic and food industry. In order to explore potential use of sugarcane wax in lipstick and cosmetic pencil formulations in place of carnauba wax, the present project “evaluation of suitability of sugarcane wax for application in cosmetics” was sanctioned.

Carnauba wax, also called Brazil wax or palm wax, is obtained from the leaves of the carnauba palm. It is known as queen of waxes and in its pure state, it usually comes in the form of hard yellow-brown flakes. Because of its hypoallergenic and emollient properties as well as its shine,

carnauba wax appears as an ingredient in many cosmetic formulas where it is used to thicken lipstick, eyeliner, mascara, eye shadow, foundation, deodorant, various skin care preparations, sun care preparations, etc.

2.0 Scope of work (Study I)

- Procurement of cosmetic grade carnauba wax from market
- Procurement of sugarcane wax from M/s Godavari
- Identification and selection of standard formulations for lipstick and cosmetic pencil
- Procurement of different ingredients to be used in formulation of lipstick and cosmetic pencil
- Preparation of lipstick and cosmetic pencil formulations using carnauba and sugarcane wax
- Modification of standard formulation to develop lipstick and cosmetic pencil based on sugarcane wax to match its performance equivalent to carnauba wax
- Comparative evaluation of lipstick and cosmetic pencil formulations based on carnauba and sugarcane wax for various physico-mechanical properties as per standard protocols (IS 9875: 1990 of lipstick and IS 9832: 2002 of cosmetic pencil)
- Compilation of results and submission of report

3.0 Literature Survey

During the agro-industrial process, a large part of the wax gets dissolved in the crude juice and gets separated as the waste during the subsequent defecation-clarification step (filter cake from sugar refinery) or distillation **[1-4].**

Sugarcane wax is a potentially valuable by-product, extraction and processing costs are relatively high, leaving a rather small profit margin **[5-7].** However, the new technologies can increase the viability of cane wax as a byproduct from press mud **[8].**

Sugarcane wax recovery from filter cake/ press mud is well documented [9] as are the relevant methods and techniques at the laboratory or industrial level [10-12]. Sugarcane wax has always been a matter of interest, due to its industrial applications, in particular in the cosmetic and pharmaceutical industry [13-15]. It is a potential substitute for costly carnauba wax which is widely used in cosmetics, food and pharmaceuticals. In addition, sugarcane wax is also a source of long chain primary aliphatic alcohols, which find applications as cholesterol-lowering products [16-18].

4.0 Work Carried Out

4.1 Lipstick

The work carried out on this project has been described in detail in this section.

4.1.1 Identification of lipstick composition: For studying the feasibility of using sugarcane wax in cosmetic formulations in place of carnauba wax, various compositions have been identified from the literature as presented in Tables 1-2.

Table-1: Lipstick Composition (Reference: 'Comprehensive book on Cosmetics- Formulation, Manufacturing and Quality control' by. P. P. Sharma; Chapter-16, pp 287-300, Vandana Publication, Delhi, 1998)

S. No.	Ingredients	Wt (%)
1	Castor oil	13
2	Isopropyl linolate	1.5
3	Isopropyl myristate	2.5
4	Beeswax	7
5	Lanolin	2

6	Ozokerite wax	6
7	Carnauba wax/sugarcane wax	5
8	Candelilla wax	8
9	Propylene glycol	4
10	Acetylated monoglyceride (solid)	6
11	Acetylated monoglyceride (liquid)	3
12	Propylene glycol mono-myristate	10
13	Bromo acids	2
14	Perfume, anti-oxidant & preservative	q.s.

Table-2: Lipstick Composition (Reference: US Patent 5093111, 1992)

S. No.	**Ingredients**	**Wt (%)**
1	Castor oil	46.5
2	lanolin	3
3	Mineral oil	3
4	beeswax	3
5	Candelilla wax	5
6	Ceresin wax	5
7	Carnauba wax/sugarcane wax	2

8	Sorbitan sesquistearate	2
9	Colorants	10
10	Fragrance	0.35
11	Preservatives	0.15

4.1.2 Raw material specification

The raw materials required for the preparation of lipstick composition as per the ingredients mentioned in Table-1 & 2 were procured. Their specifications have been given in Table-3.

Table-3: Specification of raw materials used in lipstick composition

S.No.	**Material**	**Specifications**				
		B.P. (°C)	**M.P (°C)**	**Sp. Gravity (g/cc)**	**Mol. Wt.**	**Flash point (°C)**
1	Castor oil	313	--	0.961	298	--
2	Lanolin	38	--	--	756	237
3	Mineral oil	358	--	0.8	--	--
4	Bees wax	--	63	0.958	415	204
5	Candelilla wax	--	70	0.985	--	--
6	Ceresin wax	--	74	0.916	--	113
7	Carnauba wax	--	84	0.999	--	299
8	Sugarcane wax	--	66	0.972	--	290

9	Sorbitan sesquistereate	359	--	--	1061	162
10	Isopropyl linoleate	179	--	0.87	322	191
11	Isopropyl myristate	167	--	0.85	270	--
12	Ozokerite wax	--	74	0.92	--	--
13	Propylene glycol	188		1.036	76	
14	Acetylated monoglyceride	--	--	0.96-1.1	1742	--
15	Propylene glycol mono-myristate	392	--	--	286	300
16	Bromo acid (D&C Red No. 21)	--	295	--	648	--

4.1.3 Methodology for preparation of lipstick composition

Firstly, the formulation mentioned in Table-1 was prepared. The lipstick salve prepared by following the formulation given in Table-1 was not comprable with the commercial product. Then, the formulation mentioned in Table-2 was prepared and its apperance was found to be comprable with the commercial lipstick sasmple. Hence, out of the two formulations identified, the formulation given in Table-2 (USP 5093111, 1992) was selected for study.

The lipstick composition was prepared by melt mixing of all the components as mentioned in Table-2. Firstly, the liquid components (castor oil, mineral oil and lanolin) were mixed and heated in a water bath at 90-100 °C. To this, the chosen colorant Bromo acid (D&C Red No. 21)

was added after sieving through 38 micron size (Amil Ltd.) to get a homogenous mixture. The waxes were added to this homogenous mixture and melt mixed at 90-100 °C. While adding the waxes mentioned in the composition given in Table-2, either carnauba wax was used or in its place sugarcane wax was added. In the homogeneous composition prepared after wax addition, sorbitan sesquistereate was added.

The molten mass was poured in to lipstick mold and then cooled to ambient temperature. The lipstick salve was then removed from the mold and stored for further use.

4.1.4 Designing and fabrication of mold for lipstick

For the development of lipstick salve, a mold was designed in SS-304 having length 52 mm, outer diameter 25 mm and inner diameter 14 mm with one end having tapered dimension to give shape to lipstick. A mold holder was also designed as shown in Figure-1 (a-c).

Figure-1a: Lipstick mold holder

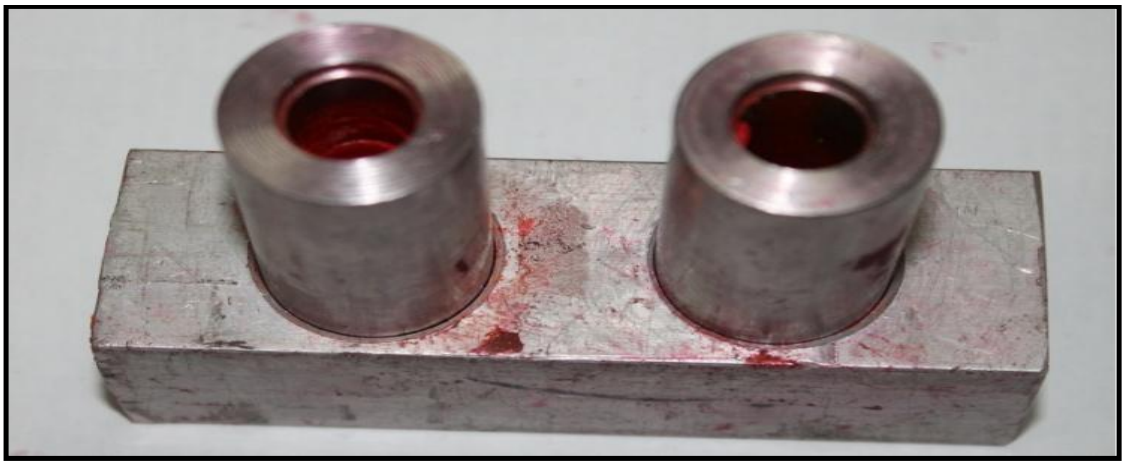

Figure-1b: Lipstick molds with mold holder

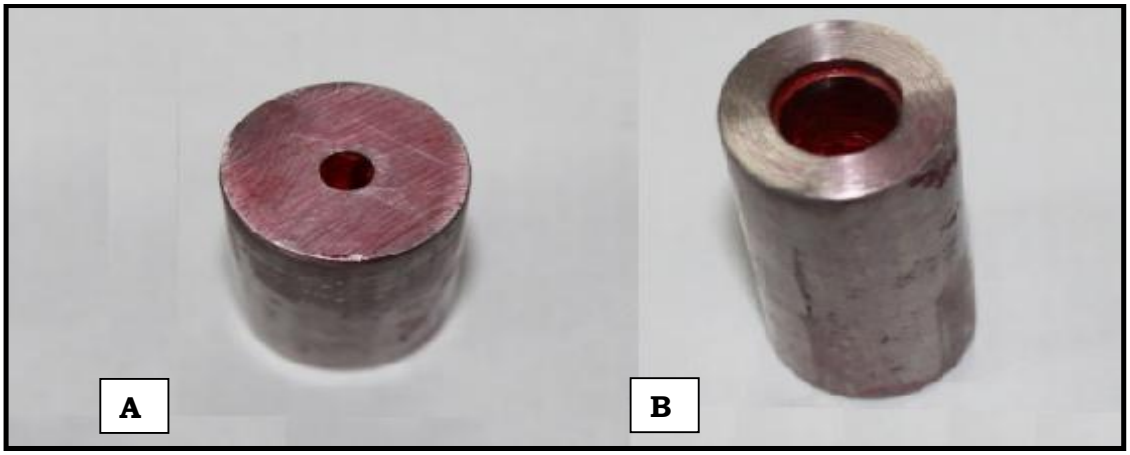

Figure-1c: Lipstick molds tapering dimensions (A) and front dimension (B)

4.1.5 Equipment used for the evaluation of lipstick salves

The characterization of developed lipstick slaves based on carnauba and sugarcane wax and the commercial lipstick sample (Lakme) was carried out as per IS 9875, 1990 (reaffirmed 2005).

a) **Softening point test apparatus:** The apparatus comprises of a controlled temperature water bath, a 12 cm long flat bottom tube having diameter 2.5 cm and 0.1°C accurate thermometer (Figure-2).

Figure-2: Softening point test apparatus for lipstick salve

b) Braking load test apparatus: This equipment measures the maximum load which a lipstick can withstand before it breaks. It comprises of a burette of 500 ml capacity held in screw chuck and an aluminum cup of 6 cm diameter and 12 cm length with an arrangement of a hook to suspend it on lipstick salve (Figure-3).

Figure-3: Breaking load test apparatus for lipstick salve

c) Pay off test apparatus: It helps in evaluating the mass release from the lipstick salve. It consists of a constant speed electric motor of power 180 watt (0.25 hp approximately) attached to a gear arrangement, which pulls the strip of paper (about 7 cm wide) from one roller on to another roller fixed on platform through supports**.** A slot arrangement having a cylindrical tube of 5.5 cm length and 2.2 cm diameter is also fixed on the platform for holding the lipstick sample (Figure-4) perpendicular to the tip of paper.

Figure-4: Pay-off test apparatus for lipstick salve

4.1.6 Characterization of Lipstick

Lipstick compositions prepared were evaluated for various properties as per IS 9875, 1990 (reaffirmed 2005).

Softening point: Lipstick was placed with protruded salve in the flat bottom tube and a thermometer was fixed through a cork in such a way that the bulb of the thermometer just touched the lipstick salve. This arrangement was placed in a 1-litre beaker filled with water such that water level was 1 cm above the upper tip of the lipstick salve and the beaker was placed in a water bath (Figure-2). Water was heated slowly while stirring so that temperature rises at a rate of 2°C per minute. When the temperature reached about 45°C, the rate of heating was decreased to 1°C per minute. The lipstick salve was observed constantly and the temperature at which the salve starts bending and losing its shape was recorded as its softening point.

Breaking load: The breaking load of the lipstick salve is the measure of its firmness and strength. The lipstick container was fixed firmly with

protruded salve of diameter ranging 11 to 13 mm, into a screw type of chuck so that the assembly was perfectly horizontal. The burette was adjusted just above the lipstick salve and a marking was made at a distance of 1.5 cm from the base of the salve where lipstick salve sited in salve holder cup (Figure-3). Weight of aluminium container was taken along with hook and suspended it on this 1.5 cm mark. Water was slowly released from the burette into the aluminium container till the salve was broken. Burette reading added with the mass of the suspended container gives the breaking load of the lipstick.

Pay off test: 1 cm portion of lipstick salve was chopped off from the tip using a sharp blade and the remaining portion of the salve was rubbed on a piece of paper to make the end portion perfectly flat. The constant speed motor was run and the time required for pulling out 100 cm of paper length was determined. The preweighed lipstick salve having flat end was inserted in the slot arrangement provided in pay off apparatus such that the flattened salve portion rested on the surface of the paper strip (Figure-4). A total load of 53 g was placed including mass of the 1ipstick. Constant speed motor was started and with the help of stopwatch, 120 cm length of paper was allowed to run. The lipstick salve was reweighed after the rub off and the length and width of the line drawn on the paper strip was measured. The pay off value was calculated using formula:

$$\text{Pay-off } (g/cm^2) = \frac{(M_1 - M_2)}{l \times b}$$

Where, M_l is the mass in gram of the lipstick before the test; M_2 is the mass in gram of the lipstick after the test; l is the length in cm of the line drawn on paper strip and b is breadth in cm of the line drawn on paper strip.

4.1.7 Requirements of lipstick

Requirements for the lipstick salve as per IS specification are given in Table-4.

Table-4: Requirements of Lipstick

S.No.	Test	Requirement	Reference
1	Softening point, *Min*	55 ºC	IS 9875: 1990
2	Breaking load value, *Min*	200 g	
3	Pay off test, *Min*	0.0001 g/cm^2	

4.2 Cosmetic pencil

4.2.1 Identification of composition for cosmetic pencil: In order to study the suitability of sugarcane wax in cosmetic pencil in place of carnauba wax, the composition mentioned in table-5 was identified from the literature for the development of cosmetic pencil.

Table-5: Composition for cosmetic pencil (Reference: EP1048285A2, 2000)

S.No.	Ingredients	Wt (%)
1	Ceresin wax	12.35
2	Bees wax	1.15
3	Carnauba / sugarcane wax	8.5
4	Propyl paraben	0.1
5	Lanolin (anhydrous)	11.5
6	Isopropyl myristate	10.0
7	Talc	8.0
8	Iron oxide (black)	3.0

4.2.2 Raw material specification

The raw materials required for the preparation of cosmetic pencil composition as per the ingredients mentioned in Table-5 were procured. Their specifications have been given in Table-6.

Table-6: Specification of raw materials used in cosmetic pencil composition

S.No.	Material	Specifications				
		B.P. (ºC)	**M.P (ºC)**	**Sp. Gravity (g/cc)**	**Mol. Wt.**	**Flash point (ºC)**
1	Ceresin wax	--	74	0.916	--	113
2	Bees wax	--	63	0.958	415	204.4
3	Carnauba wax	--	84	0.999	--	299
4	Sugarcane wax	--	66	0.972	--	290
5	Propyl paraben	--	96	1.06	180	--
6	Isopropyl myristate	167	--	0.85	270	--
7	Lanolin	38	--	--	756	237
8	Talc	--	800	2.7	--	--
9	Iron oxide (black)	--	>1000	1.5	--	--

4.2.3 Methodology for preparation of cosmetic pencil composition

The composition for cosmetic pencil was made by melt mixing of all the components as mentioned in Table-5. Firstly, the liquid portions (castor oil, lanolin and isopropyl myristate) were heated in a water bath at 90-100 °C, and to this the chosen colorant Iron oxide (black) was added to get homogenous mixture. The waxes were added to this homogenous mixture and melt mixed. Lastly, propyl paraben was added in to this homogenous mixture.

Molten mass was poured in a hollow tube of 3-4 mm diameter, cooled to ambient temperature. The cosmetic pencil was then removed from the mold and stored for further use.

4.2.4 Equipment used for the evaluation of cosmetic pencil

The characterization of developed cosmetic pencil based on carnauba and sugarcane wax and the commercial cosmetic pencil (Lakme) was carried out as per IS 9832, 2000.

Pay off test apparatus helps in evaluating the mass release from the cosmetic pencil. It consists of a constant speed electric motor of power 180 watt (0.25 hp) attached to gear arrangement, which pulls the strip of paper (about 7 cm wide) from one roller on to another roller fixed on a platform through supports**.** A slot arrangement having a cylindrical tube of 5.5 cm length and 1 cm diameter (inner) was fixed on the platform for holding the cosmetic pencil sample perpendicular to the paper sheet (Figure-5).

Figure 5: Pay-off test apparatus for cosmetic pencil

4.2.5 Characterization of cosmetic pencil

Cosmetic pencil compositions prepared were evaluated for pay off property as per IS 9832, 2000.

Pay off test: Cosmetic pencil tip was held on the paper strip kept on the machine in vertical position by a holder attached to the instrument. A load of 80.5 g was placed on the other end of the pencil. Machine was started so that the paper strip is pulled at a speed of about 5 cm/s. By doing this, a line was drawn on the paper by wearing out the pencil slip for exactly 100 cm. The cosmetic pencil was reweighed after the rub off and the length and width of the line drawn on the paper strip was measured. The pay-off was determined by finding out the wear of slip in mg/100 cm^2 area of line drawn on strip of papers.

$$\text{Pay-off (mg/100 cm}^2\text{)} = \frac{(M_1-M_2) \times 1000 \times 100}{A \times B}$$

Where, M_1 is the mass in grams of the slip before the test; M_2 is the mass in grams of the slip after the test; A is the diameter of slip in cm and B is the length of paper in cm on which the pay off test is carried.

4.2.6 Requirements of cosmetic pencil

Requirement for the cosmetic pencil as per IS specification is given in Table-7.

Table 7: Requirements of cosmetic pencil

S.No.	Test	Requirement	Reference
1	Pay off	2 mg/100 cm^2 (*Min.*)	IS 9832: 2002

5.0 Results and discussion

5.1 Lipstick

The lipstick composition prepared using carnauba wax and sugarcane wax were evaluated for softening point, breaking load and pay off. The commercial sample of lipstick of Lakme brand was also evaluated for these properties.

All the results were reported as an average of five samples for all mentioned properties. The results for softening point, breaking load and pay off test for commercial sample (Lakme), carnauba wax and sugarcane wax have been summarized in Table-8.

As evident from the results, softening points of all three lipstick samples i.e. commercial sample and the compositions made at SRI using carnauba wax and sugarcane wax were more than 55 °C, which is the minimum requirement as per IS 9875, 1990. Hence, all three samples of lipsticks are passing the test for softening point.

The breaking load values for all samples were found to be less than 200g, which is the requirement as per IS 9875, 1990. The values for SRI developed samples are much close to the required value of 200g as compared to the commercial samples.

The pay off values for all three lipstick samples was more than 0.0001 g/cm^2, which is the minimum requirement. Hence, all three samples of lipsticks were passing the pay off test.

Table-8: Summary of results of lipstick

Property	**Requirement *(Minimum)***	**Commercial sample (Lakme)**	**SRI developed using**	
			Carnauba wax	**Sugarcane wax**
Softening point (°C)	55	61.8	63.3	61.2
Breaking load (g)	200	107	193	192
Pay off (g/cm^2)	0.0001	0.00058	0.00065	0.00051

5.2 Cosmetic pencil

The cosmetic pencil composition prepared using carnauba wax and sugarcane wax were evaluated for pay off test. The commercial sample of cosmetic pencil of Lakme brand was also evaluated for the same property.

The results reported are an average of five samples tested for the mentioned property. The results for pay off test for commercial sample (Lakme), carnauba wax and sugarcane wax have been summarized in Table-9.

Table 9: Summary of results of cosmetic pencil

Property	**Requirement**	**Commercial sample (Lakme)**	**SRI developed using**	
			Carnauba wax	**Sugarcane wax**
Pay off (mg/100 cm^2)	2 (*Min.*)	2.08	2.24	2.38

As evident from the results, it was observed that the pay off tests for all three cosmetic pencil samples i.e. commercial sample and the composition made at SRI using carnauba wax and sugarcane wax were more than 2 mg/100 cm^2, which is the minimum requirement as per IS 9832, 2002. Hence, all three samples of cosmetic pencils were passing the pay off test.

6.0 Conclusion

- The preparation of lipstick and cosmetic pencil compositions has been carried out successfully by replacing carnauba wax with sugarcane wax in the compositions.
- The property profile of lipstick and cosmetic pencil compositions prepared using sugarcane wax conformed with the requirements specified in IS 9875: 1990 of lipstick and IS 9832: 2002 of cosmetic pencil specifications.
- Property profile of lipstick and cosmetic pencil prepared using carnauba wax and sugarcane wax was found to be comparable and meeting the requirements given in IS 9875: 1990 of lipstick and IS 9832: 2002 of cosmetic pencil except for breaking load of lipstick.

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