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Coconut Oil Based Low Temperature Hair Oil Formulations

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Abstract
The coconut oil based hair oil solidifies in winter. To get Solidification Point (S.P.)below 50C &good oxidative stability, the interesterification with monounsaturated fatty acid group oils was planned. Thus coconut oil on interesterification with castor oil gave product of S.P. below 50C at optimum ratio (Table 2) using immobilized enzyme from Thermo-MycesLanuginosus (TLIM),. But the lower ratio of coconut oil in this formulation led us to evolve alternative methods. The melting points of methyl esters of coconut oil &of the products of interesterified coconut oils and liquid oils suggested us to go for trans-esterification and interesterification. Trans-esterification of coconut oil with methanol using chemical catalyst was experimented as per reaction conditions of Table 3 gave Methyl esters of 2°C S.P. though viscosity was low. So, it was interesterified with coconut oil(Table 3), detailed study is underway) to improve viscosity. The interesterified Castor Oil & coconut oil was re-interesterified with MECCNO & (IE, CCNO+MECCNO) (table 4), process optimized to get product with 75% coconut oil.

Keywords: *CCNO- coconut oil, IE- interesterified, MECCNO– methyl esters of coconut oil, S.P.-Solidification point,*

I. INTRODUCTION:

Traditionally in India Coconut Oil (CCNO) is used as hair care oil by the large population. However, in winter it solidifies below about 20^0C temperature making it difficult to apply in hairs. Consideringthis it is proposed to develop a formulation so that it remains liquid in winter season. Indian market research by A.C. Neilson [1] concluded that coconut oil based hair oils comprised 2/3rd of Indian market. Its market is growing by 14% per annum.

On market analysis it was found that for winter Coconut based hair oil's famous brands are blending liquid light paraffin oil in 70-90% proportion to make the hair oil non sticky, least toxic &remain liquid [2]. Mineral oil is no way superior for the health of our hair, scalp or skin [3,4]. For coconut oil based hair oil formulation of below 5^0C solidification point to prepare without addition of mineral oil, the proper process modification is required.

Few important fat modification processes are– Fractionation, Fractional distillation of fatty acids and Re-esterification of lower molecular weight fatty acids, Inter-esterification [5], Dehydrogenation [6].Based on cost-benefit and ease of operation and multi choices the inter-esterification process is selected for study.

II. Review of the related work:

The oils which can penetrate in to the hairs can fill the gap between cuticle cells and prevent the diffusion of the low molecular weight harmful surfactants in to the hair follicle &best is coconut when compared with mineral oil & sunflower oil [7].Saturated and Mono Unsaturated Fatty Acid (MUFA) rich oils diffuse into the hair much than Poly Unsaturated Fatty Acids [PUFA] rich oils

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[5]. The Castor Oil (CO) taken with 90% MUFA[8]is also regarded as the excellent hair oil,is also used as emollient (9), along with essential oils will grow beard[10].Thus its use along with the Coconut oil is to be explored.

Reena M.B. et al. [11] conducted experiments by blending coconut oil, rice bran oil &coconut oil, sesame oil (SFA: MUFA: PUFA as 1:1:1) using 1% lipozyme IMRM of RhizomucorMiehei family for 72 hours at 37^0 C.The oil was analyzed. Based on the fatty acid composition of interesterified (IE) blends of (CCNO+RBO) & (CCNO+SESO) & their standard methyl ester's melting points, the average estimated methyl ester's melting points are calculated to be -8.7°C& -12 °C respectively.

Nagaraju A. etal. [12], studied the fatty acid compositions of the (CCNO+OLO) & (CCNO+GNO) by blending (SFA:MUFA: PUFA) in ratio (1:1:1)and of their interesterified products catalyzed by IM-60 lipase from RhizoMucormiehei. Based upon the fatty acid composition of the interesterified blend of (CCNO+OLO) & (CCNO+GNO) & their standard methyl ester's melting points, the average estimated methyl ester's melting points are expected to be -6.6 & -11.0 °C respectively.

Arita et al. [13] carried out directed interesterification of coconut oil in Acetone solvent to fractionate its oil in 3 steps at 45^0 C, 35^0 C, 25^0 C successively & and finally got 24%Medium Chain Triglycerides of10-15^0C Melting Point.

Thus interesterification route seems to be worth explored further to apply on thisresearch.Of the two oils tested for characteristics as per BIS or Alfa Laval application manual [15-17] are:

Table 1, Characteristics of oils used :

Tests	FFA	I.V.	S.V.	HV	M.P.	S,P.	Fatty Acid Composition
CCNO	1.0	9.7	263	-	25.6	22.0	C6:0-0.2, C8:0-7.0, C10:0-6.3, C12:0-47.9, C14:0-18.2, C16:0- 8.4, C18:0-2.40, C18:1-7.50, C18:2-2.0
CO	0.20	87.1	174	164	2.0		C16:0-1.02, C18:0-1.21, C18:1-3.42, C18:1&12-0H--88.26,C18:2-4.33, C18:3- 0.53, C20:1-0.40, C22:6- 0.60

III. Material & Methods:

For Esterification immobilized enzyme from Thermo-MycesLanuginosusLipozyme TLIM Make Novozymes, Denmark. The recommended parameters as per supplier, our experience with experiments are: dose 0.30-0.40% at 55° C& time 3-4 hours.

Coconut Oil was purchased from the Kanpur, U.P., India, market manufactured by Kamani Oils, Pvt. Ltd. Mumbai, India. Refined Castor Oil was obtained from Ihsedu Agro Chem Pvt, Ltd, Gujrat, India & Chemicals ExcelR grade of SD Fine chemicals along Whattman filter paper of 42 no. of G E healthcare U.K.,.Oils were weighed in laboratory's 3 digit accuracy scale in a 4 neck round bottom flask. Top B24 joint was fitted to stuffing box with stirrer, side B19 number joint attached to thermometer pocket, vacuum connection which through catch oil connected to vacuum pump via stopcock, 4th neck fitted by cork to use for sample / material addition purpose.

In 1st set of Esterification experiments the Coconut oil was mixed at 60^0 C with 6 moles Methanol and 0,30% sodium methoxide using condenser for 3 hours[14]. The products was neutralized with 0.30% phosphoric acid/ Citric acid (stoichometrically), and then settled to

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remove glycerol by draining. Then the product was hot water washed, dried under vacuum to 120^0 C & cooled to 60^0 C. The S.P. of 2°C was obtained.Rest of the interesterification experiments were carried out with 0.40% TLIM enzyme at 55^0 C for 4 hours reaction time():

TABLE NO. 2Experimental Resultsof Oils Used in Experiment:

CCNO %Ratio	CO, %Ratio	S.P. before I.E.	S.P. after I.E.	Catalyst	Time,	Temp,	RPM
100	0	22	18	0.3%NaOCH3	3 h,	60^{0}C,	90
50	50	14.5	11.2	0.4% Enzyme	3.5 h	55-60^{0}C	90
40	60	12.5	9.0	0.4% Enzyme	3.5 h	55-60^{0}C	90
30	70	12	7.5	0.4% Enzyme	3.5 h	55-60^{0}C	90
20	80	10	5.5	0.4% Enzyme	3.5 h	55-60^{0}C	90
19	81	9.4	5.4	0.4% Enzyme	3.5 h	55-60^{0}C	90
18	82	8.5	5.3	0.4%Enzyme	3.5 h	55-60^{0}C	90
17	83	8	5.1	0.4% Enzyme	3.5 h	55-60^{0}C	90
16	84	7	4.5	0.4% Enzyme	3.5 h	55-60^{0}C	90
15	85	6.5	4.0	0.4% Enzyme	3.5 h	55-60^{0}C	90
10	90	5.5	2.5	0.4% Enzyme	3.5 h	55-60^{0}C	90

The linear relation between coconut oil and Castor oil interesterification ratio in respect to solidification point is observed.

Furthercoconut oil was interesterified using enzyme catalystand Trans esterified with methanol using chemical catalyst. The CCNO's S.P.18°C and MECCNOS.P.2°C were obtained.The coconutoil: castor oil.60:40 blends (interesterified) of S.P12°C, 60% and MECCNO, S.P. 2^0 C, 40%were interesterified using TLIM enzyme. Also interesterified was coconut oil: castor oil interesterified blend of 18:84 ratio with butyl ester of coconut oil enzymatically, the result arenoted& giving satisfactory product in regard to S.P. & Viscosity:

TABLE NO.3Experimental Results of Esterification of CCNO & MECCNO, (CCNO+CO,IE) & MECCNO, (IE)

CCNO/CO %	MECCNO %	S.P. (Before IE)	S.P. (After IE)	Catalyst	Time,	Temp,	RPM
100 CCNO	0	22	18	0.4% enzyme	3.5 h	55-60^{0}C	90
0	100 MECCNO	22	2	0.4% NaOCH3	3.5 h	55-60^{0}C	90
(CCNO60:CO 40)-IE, 60%, S.P. 12º	MECCNO 40%, S.P. 2^0 C	8°C	3.0	0.4% enzyme	3.5 h	55-60^{0}C	90
(CCNO18:CO 84)-IE,25%,S.P.6	BECCNO 75%,S.P.,-2°C		2.0	0.4% enzyme	3.5 h	55-60^{0}C	90

Thus increase of coconut oil % & reductionof castor oil % informulations happened and low S.P. product was obtained of proper viscosity.Linear relation follow experiment variable.

Formulations viscosity tested of below 5° S, Psamples were in 30-50CP at 32-33°C. Linear relation follow experiment variable here too.

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5.	CO 21%, (CCNO, BECCNO, IE, 70%)	15 at 32° C	0.9586 at 30C	1.3Y,0.3R	3.0

IV. Results & Discussions:

As observed from the table no. 2, solidification point (S.P.) of coconut oil (CCNO) decreased by blending with the castor oil (CO) after inter-esterification (IE). TLIM enzyme is fixed bed, 1-3 specific, but there is acyl migration from 2-3/1 hence most of the time it's randomized, the max temp. it bears is 65°C (55-60°Cused). As observed, the desired result was achieved at about 20% CCNO & 80% CO composition. But % of CCNO is low . Hence CCNO was modified for its methyl esters whose SP was 2^0C. Then MECCNO was esterifiedwith CCNO in 50:50& 40:60 ratios, to get 7.5°C and 2.5^0C SP respectively.

The 50:50 and 60:40 ratios of coconut and castor IE oils were further esterified with Coconut and MECCNO IE oils of 40:60, 50:50 ratios and MECCNO and the results tabulated in table no.4. Thus about 75% coconut&25% castor oil product was obtained of 3-4° C S.P.Andformer's higher viscosity got lowered to near normal.

Work being carried: The interesterification with glycerol, glycol, Butanol, ethanol and methanol is in progress, results shall be compared along with with market samples.

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