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Moisture and FFA characteristics of ghee adulterated with palm oil using dry fractionation techniques

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Abstract
Ghee is an expensive product that costs three to six times the price of edible vegetable oil. Ghee is frequently contaminated with inexpensive vegetable oil (Palm oil). The chief techniques of fractionations are dry fractionation and Solvent fractionation for separating triacylglycerol based on melting temperature. Dry fractionation without solvents is preferred as a more neutral technique because it is the simplest and most cost-effective separation technique. In the current study of ghee was blended with palm oil at different levels such as 0, 5, 10, and 20 per cent respectively and such product was subjected for dry fractionation techniques. In the present study it showed that palm oil has high moisture content and FFA as compared to control ghee. Hence, there was increase in the effect of moisture content and FFA as the level of adulteration increased.

Keywords: Dry fractionation, ghee, liquid fraction, solid fraction, palm oil, moisture, FFA

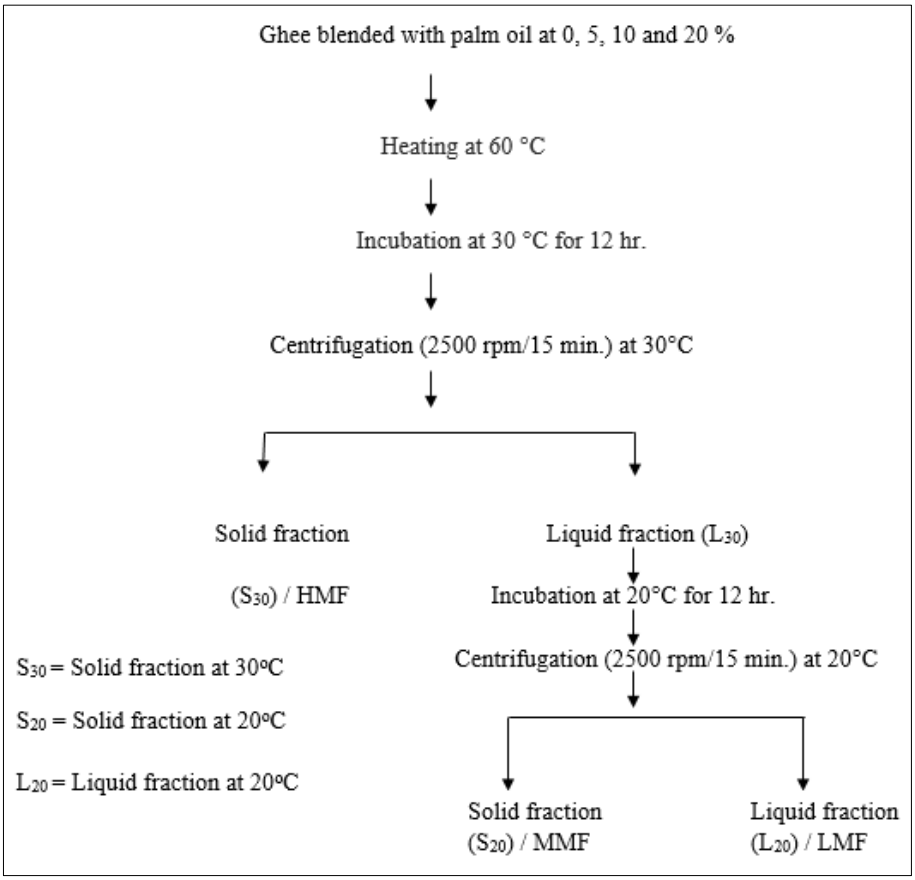
Introduction
India is the world's largest milk patron, producing 198.4 MMT and is growing at a compound annual growth rate of 6.62% compared to 827.88 MMT worldwide. The per capita availability of milk in India is estimated to reach 592 g/day by 2023-24 (Basic Animal Husbandry Statistics, 2020) [3]. Adulteration of ghee is harmful to both consumer health and the dairy industry. Unfair competition results from deceptive practises. These causes market distortions, which can harm domestic or international trade. As a result, quality testing for milk and milk products such as ghee is critical for both consumers and processors or manufacturers (Gandhi, 2014) [4]. Fractionation is the most cost-effective method for modifying the physical properties of milk fat. As a more neutral technique, dry fractionation with no solvents was preferred over solvent fractionations. The target crystallisation temperature and cooling rates were monitored at all times during the process. Temperature variations and a wide range of melting and solidification temperatures enable the extraction of fractions with varying compositions and properties (Kowalska *et al*., 2008) [8].

Ghee adulteration is most common. Ghee is frequently adulterated. Vegetable oils and animal fats are the most commonly used adulterants. Moisture is always present because boiling does not completely remove it. FFA refers to a small portion of fatty acids that are not esterified in triglycerides and are freely dispersed in the milk fat phase and slightly in the milk-water phase (FFAs). FFAs are typically present in low concentrations in milk fat, but they play an important role in the flavour of milk and milk products. FFAs are primarily formed by the action of lipolytic enzymes on glycerides (Parmar *et al.*, 2018) [12].

Materials and Methods
Materials
Butter was purchased from national brand from retail outlet near Hebbal, Bengaluru. Palm oil was purchased from the local Bengaluru market and used for blending with ghee to determine their effect on moisture and FFA ghee.

Methods
Preparation of samples
The purchased Butter was then heated on direct flame in a stainless steel vessel and clarified into ghee with continous string at a temperature of 115-117 °C. Ghee was then filtered through muslin cloths, cooled, filled in airtight glass bottles for futher analysis.

Dry fractionation technique



Kankare, 1974

These adapted with some modification with modifications. Melting method was used to fractionate ghee. The crystal memory was removed by heating ghee to 60 °C. It was then progressively cooled to 30 °C in an incubator for 12 hr. to crystallize. After centrifugation at 2500 rpm for 15 min. in a temperature-controlled centrifuge it was kept at 30 °C, the liquid was separated from the crystals by decantation. At 30°C, solid fraction obtained (S_{30}) was considered a high melting fraction. The liquid fraction collected at 30 °C was then incubated for a further 12 hr. at 20 °C. After centrifugation at 2500 rpm for 15 min. in a temperature-controlled centrifuge which was kept at 20 °C. Such produced crystals were separated. The solid portion obtained at 20 °C (S_{20}) was considered a medium melting fraction, whereas the amount that remained liquid at 20 °C was referred to as the low melting fraction (L_{20})

Moisture and FFA of palm oil, ghee and its fractions added with palm oil (S20 and L20)

Determination of moisture of ghee

Moisture of ghee was determined by the method as per IS: SP: 18 Part (IX) 1981, 5 g of ghee were accurately weighed into a previously weighed and dried aluminum dish and dish containing the ghee was heated by transferring to hot air oven at 98 ± 2 ºC for 2 hr and then cooled in a desiccator. Repeat this process and take weights every 30 min until consecutive results show not more than a 1 mg difference.

$$\text{Moisture content (\%)} = \frac{w_1 - w_2}{w_1 - w} \times 100$$

Where

W_1 = weight in g of the dish with ghee before drying, W_2 = weight in g of the dish with ghee after drying, and W = weight in g of the empty dish

Determination of free fatty acid value in ghee

Mix the melted fat thoroughly before weighing. Filter it using Whatman no. 1 filter paper to get rid of any suspended particles. Weigh accurately about 5 to 10 g of molten sample in a 250 ml conical flask and add 50 ml to 100 ml of freshly neutralized ethanol and about 1 ml of phenolphthalein indicator solution. Boil the mixture for about five minutes on a boiling water bath and titrate while hot against standard alkali solution shaking vigorously during the titration.

$$\text{Free fatty acids (as \%, O.A)} = \frac{\text{ml NaOH used X Normality of NaOH X 2.82}}{w}$$

Where

W = Weight in g of ghee sample taken

Statistical analysis

Significant difference between the values was verified by one way analysis of variance (ANOVA) and comparison between means was made by critical difference value by using R software [R. version 4.1.2 (2021-11-01), copyright © 2021, R foundation].

Result and Discussion

Moisture and FFA of palm oil, ghee and its fractions added with palm oil (S20 and L20)

The palm oil, control and fractions of ghee samples were analysed Moisture and FFA data were presented in the (Table 1, 2, 3).

Moisture content

The estimated moisture content in ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.16, 0.17, 0.18 and 0.19% respectively. Ghee with PO (20%) had higher moisture content and control ghee had lower moisture content as compared to other ghee sample which, are shown in Table 1. The moisture content not more than 0.3% in all the grades of ghee is required by AGMARK, (1987) [1]. Moisture content of maximum 0.5% as per FSSR, (2021) is legal requirement for ghee. Finding from the present work clearly demonstrated that the moisture content in ghee were directly proportional with increasing level of adulteration with palm oil (5, 10 and 20%, respectively) and Moisture content of palm oil 0.21%, respectively. It confirms with work carried out by Ekop *et al*., (2007) [5] who observed moisture content in palm oil to be 0.23%. Palm oil has high moisture content as compared to control ghee so there was increase in the moisture content as the level of adulteration increased. Pankaj *et al.,* (2013) [13] report that moisture content was 0.13% in buffalo ghee and Archana, (2019) [2] reported that ghee produced by conventional heating had 0.16% of moisture. Presences of low moisture in ghee were less than 0.5% which could be due to the high heat during clarification. The residual moisture in ghee depends on the method of preparation, heat clarification temperature and duration of heating (Mathur *et al.*, 2005) [10].

Present experiments showed moisture content in liquid fraction of ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.17, 0.18, 0.19 and 0.20%, respectively. L_{20} with PO (20%) had higher moisture content and L_{20} (Control) had lower moisture content as when compared to other ghee sample were also shown in Table 2.

The moisture content in solid fraction of ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.15, 0.16, 0.17 and 0.18%, respectively. S_{20} with PO (20%) had higher moisture content and S_{20} (Control) had lower moisture content as compared to other ghee sample were also shown in Table 3.

Table 1: Gross composition of ghee added with palm oil

Sample	Moisture (%)	Free fatty acid (% O.A)
Palm oil	0.21[a]	7.06[a]
Control ghee	0.16[a]	0.32[e]
Ghee with PO (5%)	0.17[a]	0.68[d]
Ghee with PO (10%)	0.18[a]	1.00[c]
Ghee with PO (20%)	0.19[a]	1.70[b]
CD ($P \leq 0.05$)	0.90	0.07

Table 2: Gross composition of liquid fractions of ghee added with palm oil

Sample	Moisture (%)	Free fatty acid (% O.A)
L20 (Control)	0.17[a]	0.34[d]
L20with PO (5%)	0.18[a]	0.70[c]
L20 with PO (10%)	0.19[a]	1.20[b]
L20 with PO (20%)	0.20[a]	1.90[a]
CD ($P \leq 0.05$)	1.00	0.01

Table 3: Gross composition of solid fraction of ghee added with palm oil

Sample	Moisture (%)	Free fatty acid (% O.A)
S20 (Control)	0.15[a]	0.33[d]
S20 with PO (5%)	0.16[a]	0.69[c]
S20 with PO (10%)	0.17[a]	1.10[b]
S20 with PO (20%)	0.18[a]	1.80[a]
CD ($P \leq 0.05$)	1.00	0.09

Free fatty acids

The free fatty acids content in ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.32, 0.68, 1.0, and 1.7%, respectively. Ghee with PO (20%) had higher FFA content and control ghee had lower FFA content as compared to other ghee sample were also shown in Table 1 as per FSSR, (2021) ghee may contain maximum 2.0% FFA (% O.A). According to FSSR, (2017) palm oil contains not more than 10% of FFA (% PA). Special, general and standard grade of ghee contain FFA (% O.A) not more than 1.4, 2.5 and 3.0 as given under AGMARK, (1987) [1]. The FFA content of ghee depends upon the method of manufacture of ghee, initial quality of cream or butter used for making ghee, heat clarifying temperature and the residual moisture content. Presences of moisture accelerate the hydrolysis of fats, and thereby release free fatty acids. The formation of free fatty acids is undesirable in milk fat products because the short chain fatty acids particularly butyric acids are primarily responsible for formation of rancid flavour in milk fat products like ghee. The free fatty acids (% O.A) content in ghee were directly proportional with increasing level of adulteration with palm oil (5, 10 and 20%, respectively). FFA content of palm oil was 7.06%, respectively. It confirms with work carried out by Ekop *et al*., (2007) [5] who observed a free fatty acids (% PA) content of 7.32 in palm oil and Archana, (2019) [2] who reported that ghee prepared from creamery butter produced by conventional heating process had 0.34% of free fatty acids (% O.A). Palm oil content high amount FFA may be their increase in value of FFA in ghee added with palm oil sample.

The free fatty acids content in liquid fraction of ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.34, 0.70, 1.20 and 1.90% O.A, respectively. L_{20} with PO (20%) had higher FFA content and L_{20} (Control) had lower FFA content as compared to other ghee samples which is also shown in Table 2.

The results obtained in the present study showed that FFA content of solid fraction of ghee were directly proportional to increasing level of adulteration with palm oil (5, 10 and 20% respectively). The free fatty acids content in solid fraction of ghee added with palm oil (0, 5, 10 and 20%) were found to be 0.33, 0.69, 1.1 and 1.80% O.A, respectively. S_{20} with PO (20%) had higher FFA content and S_{20} (Control) had lower FFA content as compared to other ghee sample as shown in Table 3.

Conclusions

In the present investigation the effect of Moisture and FFA of ghee adulterated with palm oil using dry fractionation techniques was studied. The ghee was blended with palm oil at various levels such as 0, 5, 10 and 20% respectively and had under gone dry fractionation. From the results it was observed that among all the samples Ghee adulterated with or blended with various levels in which L_{20} with PO had higher moisture and FFA content.

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