Journal of Stored Products and Postharvest Research Vol. 3(10), pp. 137 – 144, 16 May, 2012
Available online at http://www.academicjournals.org/JSPPR
DOI: 10.5897/JSPPR11.072
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Full Length Research Paper

Methodology assessment on melting and texture properties of spread during ageing and impact of sample size on the representativeness of the results

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Accepted 28 February, 2012

A large number of methods and instruments have been used for measuring the melting and texture properties of margarine and spread. All these methods assume that margarine or spreads are isotropic materials. Depending on the scale of the sample, such statement is sometimes questionable in particular when using miniaturized samples. This paper gives an overview of the methods adopted and evaluates its suitability to analyze the melting and textural characteristics of spreads. Differential Scanning Calorimetry (DSC) was used to analyze the melting property of spread. Textural evaluation was carried out on spread with cone penetration, creep analysis and compression test using cylinder. DSC was found to be not reproducible due to the small size of the sample; larger sample are recommended. Creep analysis by DMA was found to be a sensitive method in detecting the differences in textural attributes of spread.

Key words: Emulsion, texture, calorimetry, spread, lipid.

INTRODUCTION

Butter, margarines and table spreads are water-in-oil emulsions; they differ by the fat to aqueous phase ratio as indicated in Table 1. The type and corresponding ratio of these products may differ in different countries. Structurally, margarine consists of a continuous liquid fat phase with fat globules, crystalline fats and aqueous phase dispersed in it (Juriaanse and Heertje, 1988). The low fat spreads cannot be easily formulated to be similar to butter. Poor or slow meltability in the mouth and slow flavor release are frequently encountered difficulties in developing low fat spread products. The rheology of low-fat spread is governed by emulsion characteristics such as the proportion of the aqueous phase and the size of the water droplets (Borwankar et al., 1992). The plastic character of fat products such as margarine, shortening and butter is the result of the presence of a three-dimensional network structure of fat crystals. The rheological properties of such products can be influenced greatly by thermal and or mechanical treatments during processing (de Man, 1969). Spreadability of butter and margarine is an important aspect of the consumer acceptability of these products. The ratio of solid to liquid fat in a product is probably the most important factor determining hardness and spreadability (deMan et al., 1979). Storage temperature of the finished product is also important as a factor influencing the course of hardness changes and should, therefore, receive attention.

In the food industry, the texture of fat-containing products strongly depends on the microscopic, mesoscopic and finally macroscopic structure of the fat network formed within the finished product. The fat network provides firmness or solid-like behaviour to products such as margarine or spreads. Liquid fat surrounding the fat globule acts as a viscous fluid and flows on application of stress (Diener and Heldman, 1968). Margarine thus exhibits viscoelastic characteristics. For margarine, a very essential property in practical uses is its storage stability. During storage, the changes of physical properties are reflected by the changes in the crystals and the crystal network in the margarine (Zhang et al., 2005). The rheological characteristics of finished

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Table 1. Water-in-oil emulsions.

Type of emulsion	Country	Fat phase	Aqueous phase
Low fat spread.	US (patent 4071634).	30-50% fat phase.	Phosphatides, proteinaceous ingredients.
Low fat spread.	Europe (EP patent 0327288).	25-70% w/w of fat phase.	Non gelling starch hydrolysate.
Lactoprotein-free fat spread.	Canada (CA 2007770).	20-60% by weight.	0.1 to 5% by weight of gelatine or agar agar and 0.1 to 5% by weight of amylopectin rice starch.
Margarine.	Canada (CA 1271364).	Fat content 50-60%.	
Low-fat butter or margarine.	Canada (CA 2032337).	15 to 50% lipid.	40 to 60% moisture.
Butter.	France.	82% minimum.	18% maximum.
Butter.	UK.	80% minimum.	20% maximum.
Margarine.	Central Europe.	80% or less.	20% or more.
Margarine.	US.	80% or less.	20% or more.

margarine are expressed in terms such as consistency, texture, plasticity, hardness, structure and spreadability. Studies on the effect of storage conditions on the quality of retail margarine have a tendency to focus on the changes in the physical, chemical and rheological properties that occur during storage (Laia et al., 2000). The water droplet size distribution of fat spread is an important quality characteristic. The growth of microorganism is delayed when the water droplets in which they live are so small (<5 µm) that the amount of nutrients per droplet is insufficient (Van Dalen, 2002).

Product attributes such as spreadability, hardness and work softening are determined at least partly by the shape and size of the individual fat crystals and the way in which these fat crystals interact to form clusters, agglomerates and networks. For the fat blends cooled to 35°C, the surface area sizes ranges from 0.1 to 0.2 μm^2, approximately with an average of 0.34 μm^2. For samples cooled to 5°C, the surface area sizes ranges from 0.003 to 0.07 μm^2 with an average of 0.018 μm^2 stating that more rapid cooling leads to smaller crystals (Heertje and Leunis, 1997). Margarines and butter containing relatively large crystals (>5 µm) at high solid content are harder, more brittle and grainy than those containing small crystals. At low solid content, large crystals cannot incorporate as much liquid oil as small crystals and the product becomes oily (Chrysam, 1985). Large needle like crystals are usually beta crystals, while small ones are beta prime crystals (de Man et al., 1990). The crystal form of soft margarines were analysed by deMan et al. (1991a). The canola margarine containing only beta crystals had no surface sheen, appeared dull and crystals were large. The crystals of other margarines were small containing beta and beta prime crystals.

Different studies were made on physical and textural properties of margarine and spread since the product development. Melting characteristics of these products are important for flavour release and consumer acceptance. DSC measurements were used to quantify the melting of fat crystals in these products. For melting characteristics of Vanaspathi, a hydrogenated vegetable fat commonly used in India as a substitute for butter, the samples were stabilised according to the IUPAC method (1987) and the thermograms were recorded by heating at the rate of 2°C/min from -5 to 60°C (Jeyarani and Reddy, 2005). The DSC analysis of shortenings and margarines were carried out with a model 900 du Pont Thermal Analyzer. Heating and cooling rates were 5°C/min (de Man et al., 1989). Thermal characteristics of butter were analysed using SETARAM Micro DSC-II type ultra-sensitive scanning calorimetry (Schaffer et al., 2001). The measurements were carried out in the temperature range of 0 to 50°C with the heating and cooling rate of 0.3°C/min. Consistencies of plastic fat products are closely related with their flow properties. One of the manifestations of viscoelastic material is that they undergo creep, that is, continue to deform under constant stress or load (Purkayastha et al., 1985). Relative studies have been reported to study viscoelastic properties of plastic fat products. de Man (1985) developed a creep analysis instrument to measure strain under constant stress as a function of time to determine the elastic and viscous components of butter and margarine. The selected force range from 4.9 to 19.6 N and cylindrical

samples 2.3 cm diameter and 2.0 cm length were prepared by using stainless steel boring tube. The most common method used for evaluation of textural properties of margarine is the cone penetrometer (AOCS, 1974 method Cc 16-60). The cone is driven into the product by the force of gravity and the penetration depth is measured. Hayakawa and de Man (1982) suggested the term “hardness index” where the weight of the cone assembly is divided by the depth of penetration. Instron Universal Testing Machine (IUMM) and Ottawa Texture Measuring System (OTMS) were used in both the penetration and compression test to analyse the texture of ‘stick margarine’ (de Man et al., 1990). The texture of fat blends were analysed using a penetrometer PNR 10 equipped by a cone with the angle 40° (Unilever cone) and connected with a plunger of the total weight of 159 g (Piska et al., 2006). The aim of the present study is to assess the different techniques used for the evaluation of physical and textural properties of spread. A slight modification was made with the established reference method for melting profile and texture and the technique was assessed to determine the melting behaviour, uniformity in fat distribution and texture in the spread produced from the local company.

Results obtained from this study will be helpful in optimising the techniques used for the characteristics of spread for future studies. A secondary objective is to assess the impact of the size of the sample on the measured property and finally to decide if a sample can be representative for the selected method.

MATERIALS AND METHODS

The sample was obtained from the local company soon after production and was immediately stored at 4°C to analyse the melting and texture changes during its ageing for 4 days from production day.

Water content measurement

Water content of spread was determined using the AOAC method 925.10 (AOAC, 1996). Water content values were average of three measurements.

Thermal measurement

The thermal behaviour of spread was determined by differential scanning calorimetry (DSC Q100, TA Instruments – Waters, France). The equipment was calibrated with indium (m.p. = 156.61°C and ΔH = 28.54 J/g), water and sapphire and an empty pan was used as reference for these calorimetric measurements. About 15 to 20 mg of spread stored at 4°C was sampled from the tub of margarine and was installed in the DSC pan in a walk-in-cold chamber. Then the pan was wrapped in a thermal insulation and was quickly installed in the DSC oven. This procedure allows a better control of the cold chain of spread and permit to reduce heat up that may have a strong impact on fat crystals that have crystallised during storage. Sample was cooled down at -5°C and was equilibrated for 3 min *in situ* in the DSC oven. This short cool down was made to have a noise less calorimetric signal starting at around 5°C, which represents the lower temperature of the temperature range of interest when studying lipids in the case of eatable spreads and butter (roughly temperature of a domestic refrigerator. This rapid cooling was applied to all samples and it was assumed that it had a minimal impact on the solid content of the sample. A heating rate of 3°C/min was then used until reaching a final temperature of 50°C, followed by an isothermal plateau at 50°C for 10 min. Nitrogen gas flow of 50 ml/min was used to avoid any water condensation in the calorimeter head. The TA instruments software was used to record and analyse the thermograms.

Since the repeatability of the measured melting enthalpy was not very good, larger pans, containing 60 mg were used for the DSC tests. Melting enthalpy was determined by averaging three replicates. Calorimetric measurements method used in this study is slightly different from that used by Borwankar et al. (1992) with Perkin-Elmer DSC-7 where the samples are cooled from 4.4 to 0°C at -40°C/min, and then the heating scan was begun immediately upon reaching 0°C with heating rate at 10°C/min. In another method the samples were chilled with liquid nitrogen to -50°C before measuring the melting profiles (de Man et al., 1979). In AOCS official method Cj 1-94 (AOCS, 1995) of fats and oils, the samples (7 $\pm$ 0.2 mg) were heated rapidly and held at 80°C for 10 min, cooled to –60°C at 10°C/min and held for 30 min and then heated to 80°C at 5°C/min.

Texture measurement – Large deformations

Different techniques were tested and compared to find out methods suitable for the characterisation of the texture of spread during ageing. Texture profile analysis (TPA) was performed with a Universal Testing Machine Lloyd LR 5K (AMETEK SAS, France) equipped with a force capacity of 50 N. NEXYGEN MT Data Analysis software was used to analyse the data. A conical probe (60°) was driven at 1 mm/s to a depth of 19 mm twice. A force-time curve is obtained with this TPA test and hardness is defined as the peak force during the first compression cycle (Bourne, 1978). The samples were tested in their original containers which was a plastic pot (250 ml). The pot was installed on a solid plastic holder frame which was machined to perfectly match the geometric profile of the bottom of the spot. Such a system permits to prevent any artifact due to incorrect contact between the pot and the seat of the texture analyzer. Three replicates were done during 1, 2 and 3 days of storage. Two compression tests were performed with a Texture Analyzer LFRA (Brookfield, United States) equipped with a capacity of 10 N. LFRA software was used to analyse the data.

A first test was performed with a conical probe (40°) at 1 mm/s down to 10 mm in the sample. A second test was performed with a cylindrical probe (diameter 12.7 mm; height 35 mm) at 1 mm/s to a depth of 10 mm. The penetration force (in gram) was reported as hardness. For both tests, the samples were tested in their original containers and the test was replicated three times at ambient temperature.

Texture measurement - Small deformations

Dynamic mechanical analysis (DMA) offers a potentially interesting alternative to conventional texture tests such as those performed so far. Indeed, the sample installed in a DMA is much smaller (typically in our case a cylinder of 13 mm diameter and 8 mm height) and a much better control of the ambient temperature of the sample can be obtained with a stability of 0.1°C. Liquid nitrogen was used to refrigerate the equipment, inducing more expansive experimental conditions. The measurement of the force and of the displacement is also done with a much higher sensitivity and accuracy than with

Table 2. DSC analyses of different spots from the same tub of spread.

Spot	Melting program – I peak		Melting program – II peak		Melting program (I+II) peak
	T (°C)	Enthalpy (J/g)	T (°C)	Enthalpy (J/g)	Enthalpy(J/g)
1	16.95	1.90	24.15	2.72	4.62
2	13.71	0.55	21.44	3.10	3.65
3	12.93	1.03	22.13	3.99	5.01
4	14.12	0.49	21.66	2.93	3.42
5	13.99	0.26	22.02	3.93	4.19
6	13.92	0.21	22.19	3.20	3.41
7	13.77	0.14	20.90	3.68	3.82
8	14.77	0.88	23.66	4.28	5.16
Average	14.27±1.12	0.68±0.55	22.27±1.03	3.48±0.53	4.16±0.68

the Lloyd machine. With our equipment (TA Q800 – Waters Instruments, France), the force resolution was 0.0001 N and the strain resolution was 1 nm. Specific tests have been carried out and have been compared to texture tests. Sample preparation was done in cold chamber to avoid melting of the margarine. The sample is then insulated and immediately transferred to DMA with settings at DMA creep with preload force of 0.001 N (preload force is the force necessary to have contact between the sample and movable clamp).

The sample was equilibrated at 5°C for 10 min and stress of 0.002 MPa is applied on the sample for 10 min and recovery is observed for 20 min to determine permanent deformation. The test was conducted for 3 days with 2 replications.

RESULTS AND DISCUSSION

Uniformity analysis

Since some heterogeneous results were preliminary obtained on samples of small size, the uniformity of the spread composition within a same container (commercial container of 250 ml) was assessed. 8 samples within a single container stored at 4°C for 3 months were picked up at different locations to represent the overall volume of spread. Thermal and water content analysis were performed for each sample. The results of the uniformity of fat distribution by DSC analysis of 8 samples taken from the same tub of spread are shown in Table 2 while the thermograms are illustrated in Figures 1 and 2. Upon melting, two peaks are observed. The thermogram shows a small first peak at temperature between 12 and 14°C and a second broader peak at 20 to 24°C. The melting profile is not uniform for the 8 samples taken for the test. There is difference in the evolution of first and second peak and the melting enthalpy values. There are small differences in the temperature of first and second peaks but significant differences in the melting enthalpy values. The first peak occurs at 14.27 ± 1.12°C and the second peak at 22.27 ± 1.03°C. The melting enthalpy for the first peaks is 0.68 ± 0.55 J/g and 3.48 ± 0.53 J/g for the second peak. There was no significant difference in the water content of the 8 samples; the water content was between 39.02 and 39.23% for the 8 different samples.

Ageing analysis

Mean values of melting temperature and enthalpy of spread as function of storage is displayed in Table 3. There is a slight decrease in the peak temperature and an increase in the enthalpy of spread during ageing from production to 4 days. After 4 weeks of storage there is an increase in the melting temperature and decrease in the enthalpy of spread. The fats in the soft diet margarine and stick products melted below 40°C (Borwankar et al., 1992). DSC analyses of some North American shortening showed that at temperature around 25 to 30°C substantial amounts of solids melted in these mixtures (de Man et al., 1991b).

Texture profile analysis

The texture of fat-structured food products is strongly influenced by the structure and mechanical properties of their underlying fat crystal networks (Marangoni, 2002). In texture measurements it is important not to disturb the integrity of the crystal network when sampling the material (de Man et al., 1990). In this study, all the texture measurements were made with the original containers as supplied by the company. By ‘texture profile analysis’, a force time curve is plotted and analyses of the force-time curve led to the extraction of different texture parameters namely, hardness, cohesiveness, adhesiveness, springiness, gumminess and chewiness (Bourne, 1978). Textural attributes of margarine analysed during the study using TPA test (Lloyd) is displayed in Table 4. Mean value for hardness for the margarine for day 1, 2 and 3 were 15.05, 15.31 and 15.38 N, respectively. There is not much difference in the texture during the post crystallisation period. The increase in hardness is less pronounced, as the crystallisation of margarine is essentially complete during the production process and

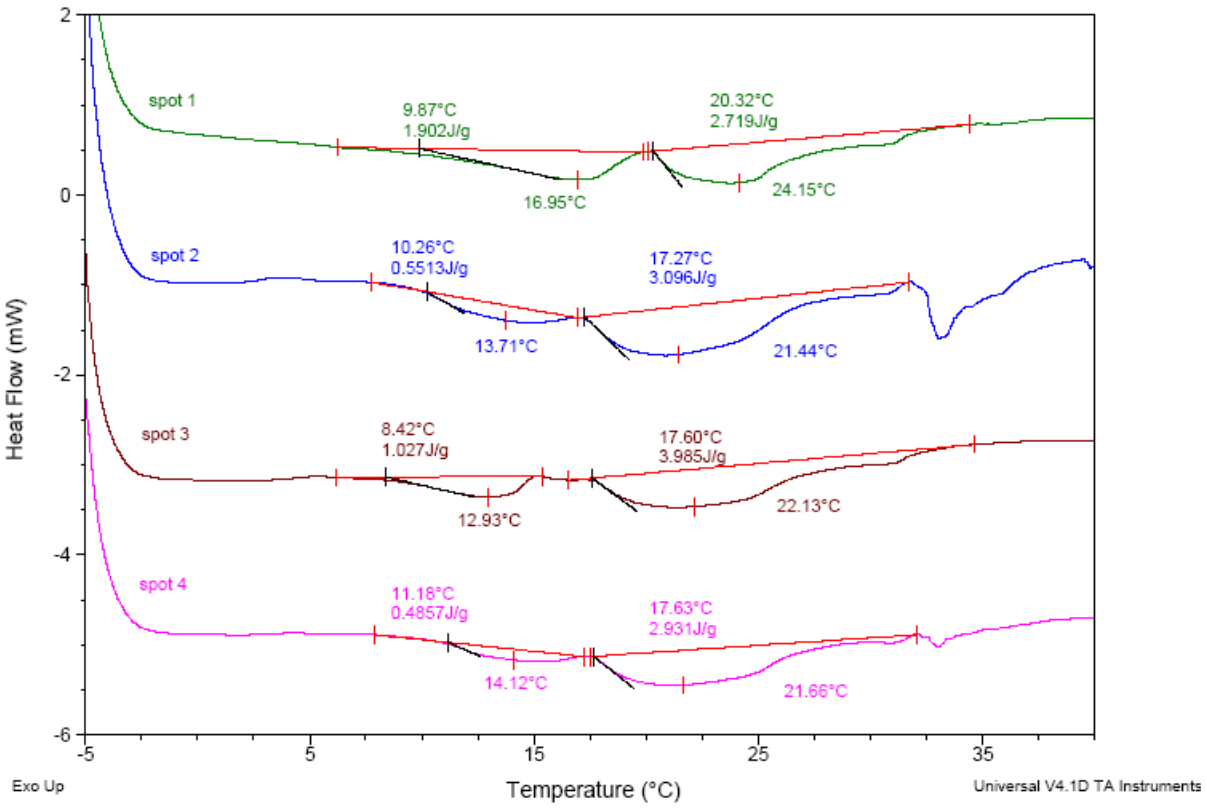

Figure 1. DSC thermogram of spot 1 to 4 taken from the same tub of spread; a relatively large dispersion of the melting enthalpy (in J/g) and of the peaks temperatures is observed even though the sample was taken from the same tub. The sample mass for the DSC was 15 to 20 mg.

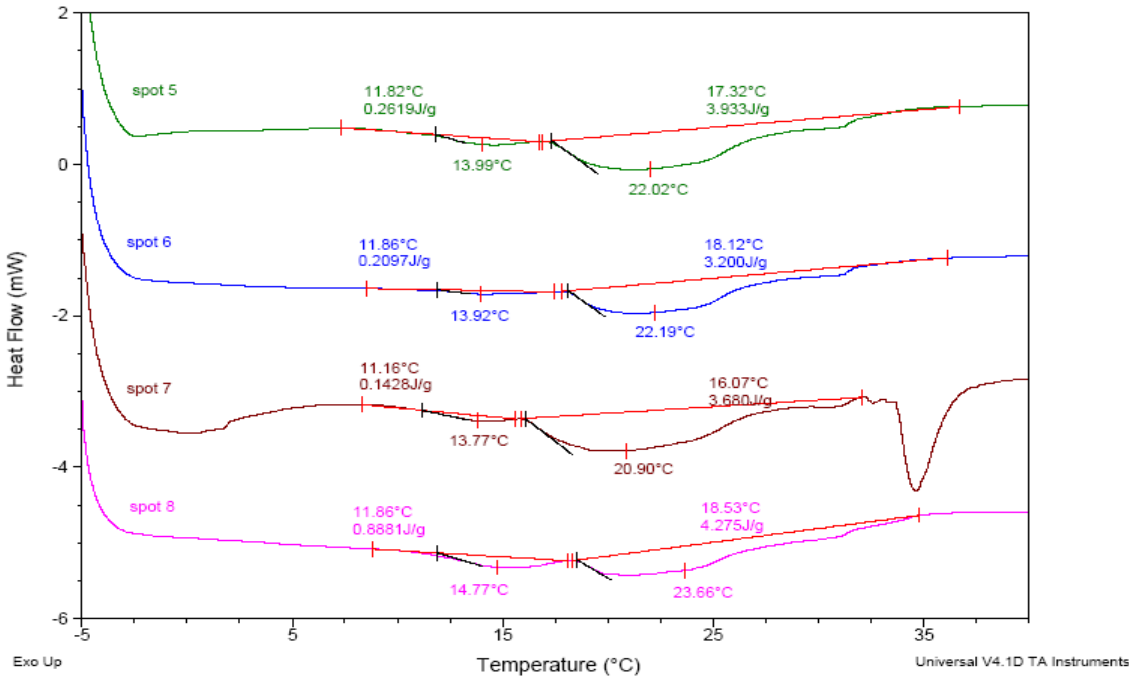

Figure 2. DSC thermogram of spot 5 to 8 taken from the same tub of spread; a relatively large dispersion of the melting enthalpy (in J/g) and of the peaks temperatures is observed even though the sample was taken from the same tub. The sample mass for the DSC was 15 to 20 mg.

Table 3. Mean value of melting temperature and enthalpy on the DSC thermograms of spread during storage.

Storage period	Melting temperature (°C)	Melting enthalpy (J/g)
1 day	23.10	5.45
2 days	22.24±0.01	7.15±0.04
4 days	21.94±0.00	8.50±0.03
After 4 weeks	24.75±0.02	6.76±0.21

Table 4. Texture profile analysis of spread evaluated using Lyold testing equipment.

Days	Hardness (N)	Adhesiveness (J)	Cohesiveness	Elasticity	Gumminess (N)	Chewiness (N)
1	15.05±0.66	0.012±0.003	0.301±0.035	0.321±0.027	4.53±0.54	1.46±0.27
2	15.31±1.08	0.015±0.002	0.337±0.035	0.325±0.021	5.13±0.27	1.67±0.19
3	15.38±0.32	0.015±0.001	0.351±0.015	0.345±0.026	5.53±0.28	1.86±0.22

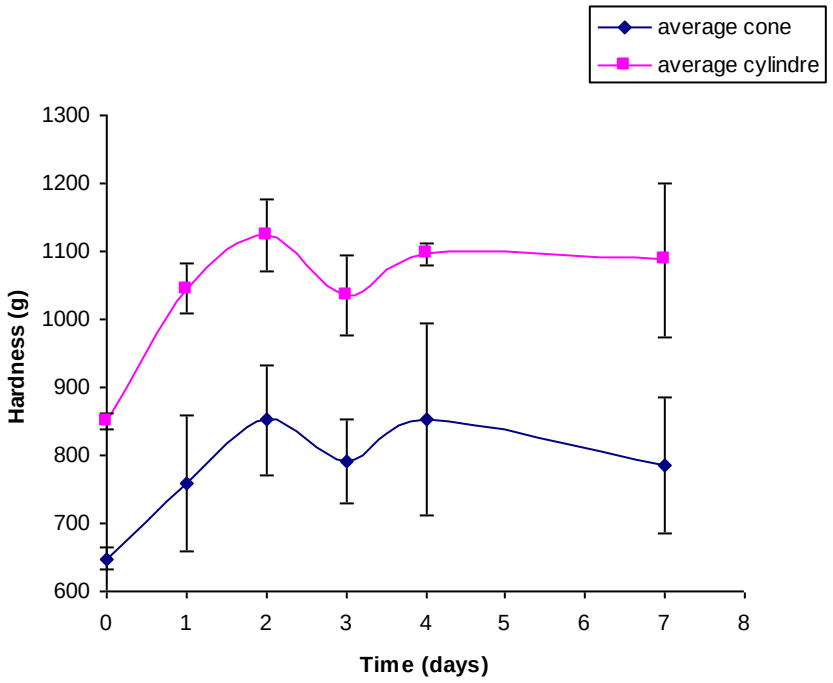

Figure 3. Texture of spread analysed using cone and cylinder during ageing (LFRA).

the crystal particles sets in rigid structure texture within 9 to 12 h of production. In studies by de Man et al. (1990 and 1991), mean value for hardness, penetration and compression for soy-bean stick margarines at 10°C were 15.6, 11.0 and 2.5 N and 8.5, 6.5 and 1.56 N for soybean soft margarines at 5°C while those for canola stick margarines were 17.1, 16.0 and 5.3 N at 10°C and for canola soft margarines were 7.3, 4.7 and 1.13 N at 5°C, respectively.

The changes in the hardness of margarine determined using cone and cylinder using LFRA texture analyser during post crystallisation period are displayed in Figure 3. An increase in hardness is found until the first 2 days during ageing and thereafter a wide variation in texture was observed in both techniques used for analysing the texture. The results of the two techniques used for determining the texture shows the same trend in hardness of margarine during ageing. The texture of shortenings and margarines were analysed by cone penetrometer and compression (de Man et al., 1989). The margarines, although similar in solid fat content exhibited different textural properties and hardness values showed the same trend as Instron results. In this study, the texture analysis was done at 20°C whereas the

Table 5. Creep analysis of spread using DMA (mean value).

Storage period (days)	Initial deformation (%)	Recovery (%)	Permanent deformation (%)
1	1.69±0.34	0.58±0.16	1.12
2	1.86	0.81	1.05
3	3.50±0.61	1.01±0.00	2.49

Table 6. Representation of sample for DSC and texture by different authors.

Reference	Sample size	Measurement	Comments
de Man et al. (1979)	10-16 mg	DSC	The fats of the soft margarine have different thermograms with melting area in the 10-30°C range.
de Man et al. (1991)	25 x 16 mm	Penetration test – texture	Compression test is a sensitive method. Correlation of values within the textural method was significant.
	Cylindrical sample 20 x 20 mm	Compression test	
AOCS Official method (2000)	7±0.200 mg	DSC of fats and oil	DSC melting properties of fats and oils.
Laia et al. (2000)	Cone angle at 40, penetration depth in 0.1 mm	Hardness-cone penetrometer	ANOVA analyses showed no significant difference in the hardness of all samples during storage.
Schaffer et al. (2001)	700-900 mg	DSC	Melting of butter fat.
Campos et al. (2002)	5-10 mg	DSC	Melting behaviour of milk fat and lard.
Campos et al. (2002)	2 x 3.4 cm cone angle at 50.3	Hardness-cone penetrometer	Samples crystallised rapidly have higher values than those crystallised slowly.
Jeyarani and Yella (2005)	15 mg	DSC	Melting characteristics of Vanaspathi.

sample was initially stored at 4°C. In the case of TPA tests (cone), the cone was at 20°C which may induce an artefact due to the heat transfer between the cone and the spread. This could partially explain the higher variability observed in the TPA tests.

Creep analysis

Creep analysis is done at small deformations and involves none or minimal structure breakdown. The creep curves of a plastic fat represent the relationship of strain and time at constant stress (de Man et al., 1985). The results of the deformation during ageing of spread for 3 days are shown in Table 5. From the results it was observed that permanent deformation increases with storage period and so it implies that the product becomes less elastic with time of storage period. This study permitted to point out the importance of the size of the sample and the difference which may be observed when using different methodology to assess the texture and the melting enthalpy of lipids in the case of eatable emulsions (spread, butter). The production of spread and butter is made under the shearing combined to refrigeration (scrapped heat exchanger). The solid content (crystallised lipids) is also known to change during storage. Therefore, cluster of recrystallized lipids may be present in the emulsion, which looks uniform and isotropic, but which is relatively non isotropic. This being said, it is obvious that the size of the sample and that the methodology used may induce some artefact. This is especially the case with modern equipment for which the trend is often to miniaturize the sample.

Crystallisation and melting in water-in-oil emulsions is a complex phenomenon affected by many factors such as emulsion droplet size, droplet-droplet interaction, polymorphism, effects of cooling rate and subsequent temperature variation (Campos et al., 2002). The size of the sample for various measurements represented by different authors in their studies is given in Table 6. Different authors have used different sample size for DSC and texture measurements and from Table 6 it is difficult to assume which sample size can be taken as representative for particular analysis. There should be a representative sample size which is suitable for each specific analysis. From our DSC tests on the uniformity of

Table 7. Recommendation on the sample size for DSC and texture (TPA test).

Size - scale	10 cm	1 cm or 1 cm/1 g	0.1 cm or 10 to 100 mg
Adapted	Texture - TPA	Melting enthalpy DSC	-
Fair		Texture - DMA	Melting enthalpy - DSC
Risky		Texture – TPA	Melting enthalpy - DSC
Not concerned	DSC texture – DMA	-	Texture – TPA; Texture - DMA

spread composition and melting behaviour of spread during ageing, recommendation can be made to take the sample of 60 to 65 mg (or more) from the centre of the tub of margarine and also from the same sample for each analysis. Additional recommendations for DSC and texture analysis are discussed in Table 7.

Conclusions

Results of the different methods adopted to assess melting behaviour and texture of spread shows its suitability to analyse shows that sample preparation method should be carefully considered for DSC and DMA as it can influence on the variability on the experimental results. Since the crystallisation of lipids in an emulsion is not isotropic, it is desirable to use large pans for the DSC tests containing about 65 mg of the product or more. Compression test using cylinder with LFRA texture analyser can be used to produce reproducible results at room temperature. The texture of spread depends on solid content of fat, crystal network, number of crystals as well as their size, morphology and polymorphism. Hence, future work is being carried out to analyse the above mentioned factors and to relate these structural characteristics to the rheological properties of spread.

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