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ORIGINAL PAPER

Physicochemical, Textural and Viscoelastic Properties of Palm Diacylglycerol Bakery Margarine During Storage

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Abstract Physicochemical, textural and viscoelastic properties of palm diacylglycerol (PDG) bakery margarines (DOS720, DOS721 and DOS711) and commercial margarine (CM) throughout a 3-month storage period were evaluated and compared. All the margarines had significant ($P < 0.05$) increments in slip melting point (SMP), solid fat content (SFC) and hardness during storage with CM having the highest overall increment followed by margarines DOS711, DOS 721 and DOS720. The smaller increments are mainly due to the ability of PDG to delay polymorphic transformation from β' to β form. In terms of viscoelastic properties, all margarines had a higher degree of firmness which may probably be due to rearrangement of the fat crystals into a three-dimensional scaffolding network upon storage. In terms of melting behavior, storage has no effects on all margarines with the exception of margarine DOS711. The melting behavior of margarine DOS711 displayed a probability of oil exudation during storage. As for polymorphic transformation, CM had the earliest polymorphic transformation with only β crystals after 8 weeks of storage. PDG bakery margarines managed to retard the transformation to more than 10 weeks of storage for DOS711 and 12 weeks of storage for DOS720 and DOS721.

Keywords Palm diacylglycerol · Palm olein · Palm stearin · Margarine · Physicochemical properties · Textural properties · Viscoelastic properties · Storage properties · Polymorphism

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Introduction

Margarine is a water-in-oil emulsion in which water droplets are kept separated by fat crystals [1]. Since its first invention, margarine has undergone exceptional development and innovation. The product range includes table margarine, bakery margarine, specialized puff pastry margarine and has now been extended to various low calorie spreads which essentially contain much higher levels of water and lower levels of fat than those legally required in margarines [2].

Bakery margarine, when used for cookies, imparts a variety of beneficial properties to the cookie dough and to the finished cookies such as lubrication and aeration of the dough, it aids in flavor release and provides desirable texture and mouthfeel to the cookies [3]. For optimum baking performance, bakery margarine should contain a minimum of 10% solid fat content (SFC) at 20 °C to prevent oil exudation or oiling off. In addition, it should also contain a minimum of 8% SFC at working temperature (25 °C) to withstand dough making [4]. In addition, it should have a high consistency or firmness without the need for refrigeration. Another important characteristic of bakery margarine is crystallization in the form of β' in order to produce cookies with a crispier and better snap texture [3].

In the formulation of margarine, diacylglycerol (DAG) is sometimes present in small quantities as an emulsifier. Although with promising health values such as decreasing postprandial lipidemia, preventing and managing obesity [5], incorporation of DAG as a main component in the fat phase of margarine formulation has not been common up till the present. In fact, studies on the effects of DAG incorporation on functional properties of margarine are limited. To the authors' knowledge, there is only one such study by Sikorski [3]. Sikorski [3] found DAG-enriched bakery margarine provided lubricity and flow ability to the dough, hence yielding increased spread in cookies. DAG-enriched margarine also allowed higher water retention during baking which led to higher gluten development. Higher gluten development changed the texture of cookies from snap type to more of a soft batch type which increased the firmness of the cookies.

As in shortening, careful consideration should be given to the storage stability of margarine. During storage, changes may occur to physicochemical, textural and viscoelastic properties of the margarine. This is mainly due to changes in the crystals and crystal network of margarine. Changes in the crystals and crystal network often lead to deterioration of margarine quality such as oiling-off and development of a sandy taste [6]. According to Wright and Marangoni [7] and Siew and Ng [8], DAG has the ability to stabilize metastable polymorphs in fats. With the retardation in transformation of crystals and crystal network, storage stability of DAG-enriched margarine will be increased. At present, any literature on the storage stability of DAG-enriched margarine has not been found. Thus, efforts should be made to understand further the effect of DAG incorporation on the storage stability of margarine.

This study aimed to evaluate the effect of three months storage on physicochemical, textural and viscoelastic properties of palm diacylglycerol (PDG) bakery margarines and compare them with those of commercial margarine (CM). Three PDG bakery margarines were firstly developed from PDG, palm olein (POo) and palm stearin with an iodine value of 44 (PS IV44). They are known as margarines DOS720 ($X_{PDG} = 0.7$, $X_{POo} = 0.25$, $X_{PS} = 0.05$), DOS721 ($X_{PDG} = 0.7$, $X_{POo} = 0.20$, $X_{PS} = 0.10$) and DOS711 ($X_{PDG} = 0.7$, $X_{POo} = 0.15$, $X_{PS} = 0.15$), respectively. Based on our previous work, PDG bakery margarines produced from the aforementioned formulations had high structural complementarity, ideal mixing behavior, good emulsifying properties and also crystallization in a mixture of β and β' forms (submitted for publication). Thus, it is important to study the storage stability of these margarines. PDG bakery margarines produced were stored at room temperature (25 °C) for a period of 3 months. Every two weeks from the initial week after manufacturing till the final weeks of storage, physicochemical, textural and viscoelastic properties of all the margarines were analyzed.

Materials and Methods

Materials

Palm olein (IV 56) and palm stearin (IV 44) were provided by Golden Jomalina Sdn. Bhd. (Tanjung Panglima Garang, Selangor, Malaysia). Commercial immobilized lipase from *Rhizomucor miehei* (Lipozyme RMIM) was obtained from Novozymes A/S (Bagsvaerd, Denmark). Commercial palm-based bakery margarine (CM) (Planta, Unilever) was obtained from the Giant Hypermarket (Petaling Jaya, Selangor, Malaysia). All chemicals and solvents used were either of analytical or high-performance liquid chromatography (HPLC) grade, respectively and were obtained from Sigma-Aldrich (Sigma-Aldrich, Malaysia).

Methods

Sample Preparation

PDG was produced by partially hydrolyzing POo using Lipozyme RMIM in a 10-kg scale packed bed bioreactor and purified by short path distillation as described by Cheong et al. [9]. Three PDG bakery margarines were first developed from PDG, POo and PS IV44. They are known as margarines DOS720 ($X_{PDG} = 0.7$, $X_{POo} = 0.25$, $X_{PS} = 0.05$), DOS721 ($X_{PDG} = 0.7$, $X_{POo} = 0.20$, $X_{PS} = 0.10$) and DOS711 ($X_{PDG} = 0.7$, $X_{POo} = 0.15$, $X_{PS} = 0.15$), respectively. The margarine fat phase (PDG, POo and PS IV44) was first heated at 60 °C and mixed using a motorized mechanical stirrer for 2 min to ensure homogeneity. The aqueous phase composed of water, salt, coloring and flavoring was then added to the fat phase and mixed using a motorized mechanical stirrer until the emulsion was formed. Formation of the emulsion was indicated by the presence of a single creamy milky phase with no clear visual separation. The emulsion was

Table 1 Formulation for preparation of PDG bakery margarines

Ingredient	Content (% weight)
Fat phase	80
Aqueous phase	
Water	17.967
Coloring	0.003
Flavoring	0.03
Salt	2

crystallized by mixing manually in a beaker cooled in an ice bath. The formulation for PDG bakery margarines is shown in Table 1. The PDG bakery margarines were then stored at room temperature (25 °C) for 3 months. Every two weeks from the initial week after manufacturing till the final weeks of storage, the physicochemical, textural and viscoelastic properties of all the PDG bakery margarines were analyzed.

Slip Melting Point (SMP)

The SMP for the PDG bakery margarines and CM were determined using the AOCS Method Cc 1-25 [10]. Duplicate measurements were obtained.

Solid Fat Content (SFC)

The SFC for PDG bakery margarines and CM were determined using a Bruker Minispec mq 20 pulse nuclear magnetic resonance (pNMR) instrument. The samples were first heated to 60 °C for 30 min, followed by chilling at 0 °C for 90 min and then kept at the desired temperatures for 30 min prior to measurements. The melting, chilling and holding of the samples were carried out in pre-equilibrated thermostated baths. The SFCs were measured within the temperatures ranges from 0 °C to 60 °C in 5 °C increments. Data were reported as averages of two measurements.

Differential Scanning Calorimetry

A Perkin-Elmer DSC-7 calorimeter (Norwalk, CT) was used to analyze the thermal properties of the PDG bakery margarines and CM. Samples weighing between 6 and 10 mg were sealed in aluminum pans. The prepared pans were first heated to 80 °C for 15 min to ensure that no residual nuclei remained. To obtain the crystallization curve, the samples were then cooled from the melt (80 °C) at 5 °C/min to -60 °C. To obtain the melting curve, the samples were equilibrated at −60 °C for 15 min before heating the samples to 80 °C at 5 °C/min. All samples were analyzed in duplicate.

Texture

The hardness of the PDG bakery margarines and CM were measured using a TA-XT2 texture analyzer (Stable Micro Systems Ltd). The hardness values of the margarines were measured at room temperature (25 °C) which was also the storage temperature using a cone angle of 45 °C with a penetration depth of 5 mm and a penetration speed of 1 mm/s. Double determinations were performed.

Viscoelasticity

Viscoelasticity of PDG bakery margarines and CM were determined according to the method described by Lai et al. [11] using a Haake RS 100 rheometer (Haake GmbH, Karlsruhe, Germany). The rheometer has a stabilized low-inertia air-bearing and also a high resolution digital encoder. The temperature control of the rheometer was maintained by a Haake F3 circulator waterbath with an accuracy of ± 0.02 °C. First, the samples were carefully scooped out with a flat-based plastic spatula to minimize sample deformation. It was then conditioned to test temperatures (25 °C) before measurements were taken. A parallel plate geometry (PP35) was used to perform a stress sweep at 1 Hz to determine the linear viscoelastic region (LVR). Small frequency sweeps from 0.01 to 100 Hz were then carried out within the LVR to avoid the destruction of the samples. The values for storage modulus (G'), loss modulus (G'') and Tan Delta were obtained. All samples were analyzed in duplicate.

FTIR Analysis

Subcell packing and polymorph of the PDG bakery margarines and CM were determined using the Bruker Alpha FT-IR Spectrometer according to the method described by Piska et al. [12]. The samples were sandwiched in between the potassium bromide windows and placed in the cell holder. The spectra were recorded with a total of 256 scans at a resolution of 16 cm^{-1}. A single band appearing at the infrared CH_2 rocking region r(CH_2) of 721 cm^{-1} or the scissoring region $\delta(CH_2)$ of 1,467 cm^{-1} indicated the hexagonal subcell packing; hence, the α crystalline form. In the β' form (orthorhombic perpendicular), two bands were observed at the r(CH_2) of 720 cm^{-1} and 726 cm^{-1} or the $\delta(CH_2)$ of 1,465 and 1,473 cm^{-1}. As for β form, a single band appearing at the r(CH_2) of 717 cm^{-1} or the $\delta(CH_2)$ of 1470 cm^{-1} indicating presence of triclinic parallel subcell [13, 14]. Duplicate measurements were obtained.

Results and Discussion

Slip Melting Point

Figure 1 shows changes in the SMP of different margarines throughout the 12-week storage period. All the margarines had increased an SMP during storage with CM having the highest overall increment (2.5 °C) followed by margarines DOS711 (2 °C), DOS 721 (2 °C) and DOS720 (1.75 °C). Increments in the SMP of the margarines are due to the polymorphic transformation from the β' to the β form. Food systems with β-form crystals are usually firmer with a higher SMP due to the stronger crystal network required to link the large-needle like crystals [15]. PDG bakery margarines had smaller increments in SMP due to the ability of PDG to retard undesirable polymorphic transformation [7, 8]. Similar findings were also found in our previous work which showed that PDG bakery shortenings had smaller increments in the SMP during storage as compared to commercial shortening (submitted for publication). Among the PDG bakery margarines, it is worth noting that PDG bakery margarines with a lower PS content displayed a smaller increment in SMP. This is not surprising as PS content is frequently associated with the SMP increment of solid fat products.

Tukey paired comparison was used to examine the differences in SMP for all the margarines during storage. All the margarines had significant ($P < 0.05$) increments in SMP throughout the 12 weeks storage period. Although the presence of PDG in the margarine formulation led to smaller increments in SMP, the increments are still significant ($P < 0.05$). Significant increments in SMP of PDG bakery margarines during storage may due to the presence of PS in the margarine formulation. PS which contains a large amount of saturated fatty acids such as palmitic and stearic acids is frequently associated with post-hardening during storage [16].

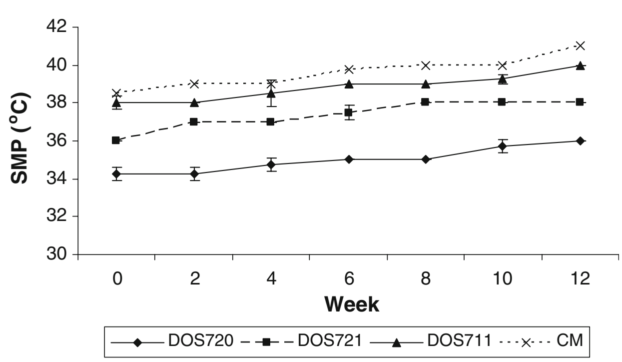

Fig. 1 Changes in SMP of PDG bakery margarines (DOS720, DOS721 and DOS711) and commercial margarine throughout the storage period of 12 weeks

Solid Fat Content

Solid fat content (SFC) is responsible for many properties of margarine including general appearance, organoleptic properties, ease of spreadability and oil exudation. At 20 °C, bakery margarine should contain a minimum of 10% SFC to prevent oil exudation or oiling off. In addition, it should also contain a minimum of 8% SFC at working temperature (25 °C) [4]. Figure 2 shows the changes in SFC for all the different margarines at 20 °C, 25 °C and 37 °C. All the margarines had increased SFC at end of the storage period with CM having the highest overall increment followed by margarines DOS711, DOS721 and DOS720. This is in agreement with previous discussion on increment of SMP. CM which had significantly ($P < 0.05$) higher SFC also had significantly higher ($P < 0.05$) SMP as compared to PDG bakery margarines.

Tukey paired comparison shows all the margarines underwent significant ($P < 0.05$) increments in SFC at the end of the storage weeks. Although PDG bakery margarines (DOS720, DOS721 and DOS711) too had significant ($P < 0.05$) increments in SFC at the end of the storage weeks, their increments were smaller as compared to CM. This is due to the ability of PDG to retard polymorphic transformation from β' to β crystals [7, 8], hence, preventing the agglomeration of fat crystals and smaller increments in SFC. This finding is in accordance with the findings in our previous work with PDG bakery shortening which showed not only smaller but also insignificant increments in SFC as compared to commercial shortening (submitted for publication).

Differential Scanning Colorimetry (DSC)

Figure 3 shows the melting curves of all the different margarines throughout the 12 weeks storage period. In order to study the melting behavior of margarines at their actual state, crystalline fats were not isolated from the margarines. Moreover, the presence of other ingredients such as water, milk, salt, coloring and flavoring too may affect the melting behavior of margarines.

One can see margarines DOS720 (Fig. 3a), DOS721 (Fig. 3b) and CM (Fig. 3d) had very similar melting behavior during storage. The number of peaks, melting peaks temperatures and endotherms sizes had no significant changes at the end of storage weeks. Thus, storage had no significant effects on the melting behavior of margarines DOS720, DOS721 and CM.

Margarine DOS 711, on the other hand, displayed different melting behavior throughout the storage period (Fig. 3c). During the early weeks of storage (0–4 weeks), margarine DOS 711 had three melting peaks in low melting fraction (LMF), one melting peak in medium melting

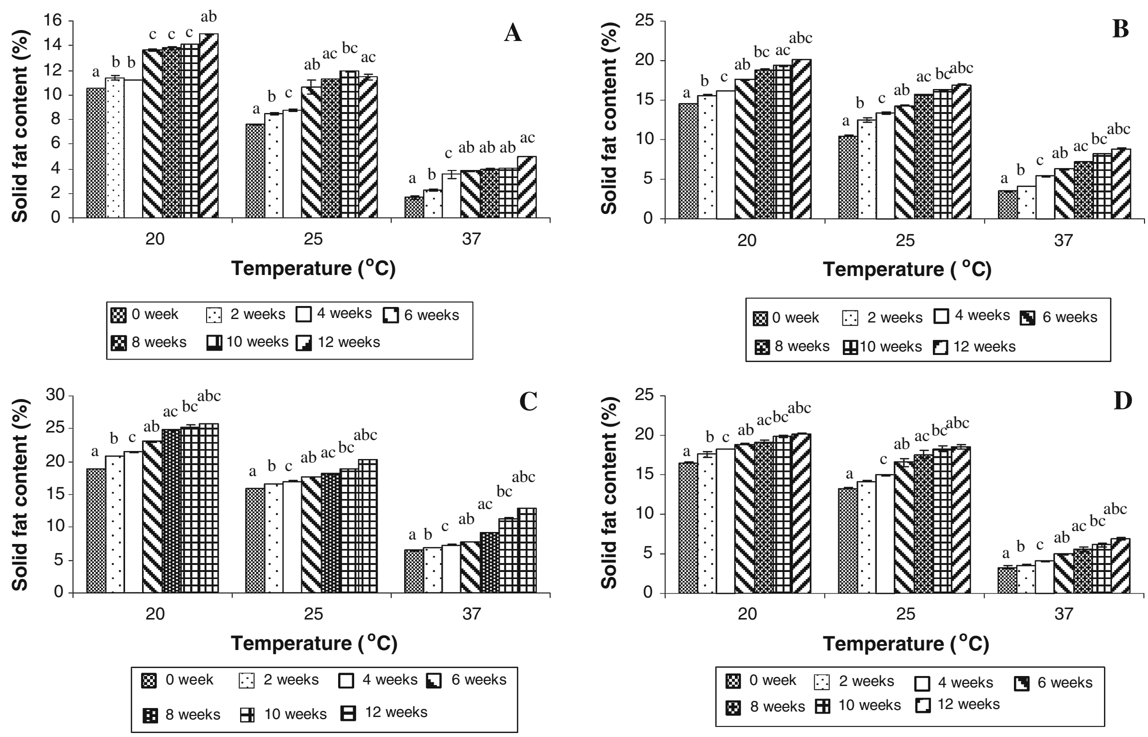

Fig. 2 Changes in SFC of different margarines at 25 °C, 37 °C and 40 °C throughout the storage period of 12 weeks **a** Margarine DOS720, **b** Margarine DOS721, **c** Margarine DOS711 and **d** Commercial Margarine

fraction (MMF) and another two melting peaks in high melting fraction (HMF). As the storage weeks proceeded from 4 to 8 weeks, a new melting peak appeared in the LMF. Appearance of this melting peak may probably due to the exudation of the lower melting fat fractions from the bulk fat phase. In fact, the melting peaks in LMF became even more prominent towards the late storage weeks (8–12 weeks). Thus, storage may led to problems of oil exudation in margarine DOS 711. Oil exudation or phase separation which refers to migration of the liquid oil phase out of the solid crystal network indicates a possibility of polymorphic transformation in margarine DOS 711. This is reflected in the following discussion on FTIR analysis of the different margarines.

Hardness and Viscoelasticity

Post-hardening is one of the problems that occurs during margarines storage. The ability of PDG to retard polymorphic transformation from β' to β crystals may be able to alleviate the problem of post-hardening in margarines. Figure 4 shows the changes of hardness in different margarines throughout the 12 weeks storage period. All the margarines had significant ($P < 0.05$) increments in hardness at the end of storage weeks with CM having the highest overall increments (12.95 g) followed by margarines DOS711 (11.49 g), DOS721 (10.81 g) and DOS720 (8.13 g). Although PDG bakery margarines had significant ($P < 0.05$) increments in hardness at the end of storage weeks, these increments are significantly ($P < 0.05$) smaller as compared to CM.

A stress sweep was firstly performed to determine the LVR of all different margarines. Knowledge of LVR is important as further analysis of viscoelastic and rheological properties were conducted within these regions in which the sample is at rest and in its actual structure. PDG bakery margarines had a larger LVR as compared to CM (Data not shown). Similar findings were found in our previous work with PDG bakery shortenings that also had a larger LVR as compared to commercial shortening (submitted to publication). This indicates that PDG had the ability to elongate fat samples to a greater extent without rupturing.

A frequency sweep was then performed within the LVR of margarines in order to examine the viscoelastic properties of the margarines in their actual state. Figures 5, 6 and 7 show the values of G', G'' and tan δ of different margarines. Figure 5 shows that the values of G' for all margarines increased with frequency, meanwhile, Fig. 6 shows values of G'' for all margarines decreased with frequency. Frequency dependency of all margarines indicates margarines had viscoelastic solid-like behavior. Increasing G' and decreasing G'' with frequency shows a transition from a more viscous to a more elastic food system. In addition, for all margarines, the values of G' were higher than G'' at any given frequency. This is displayed in Fig. 7

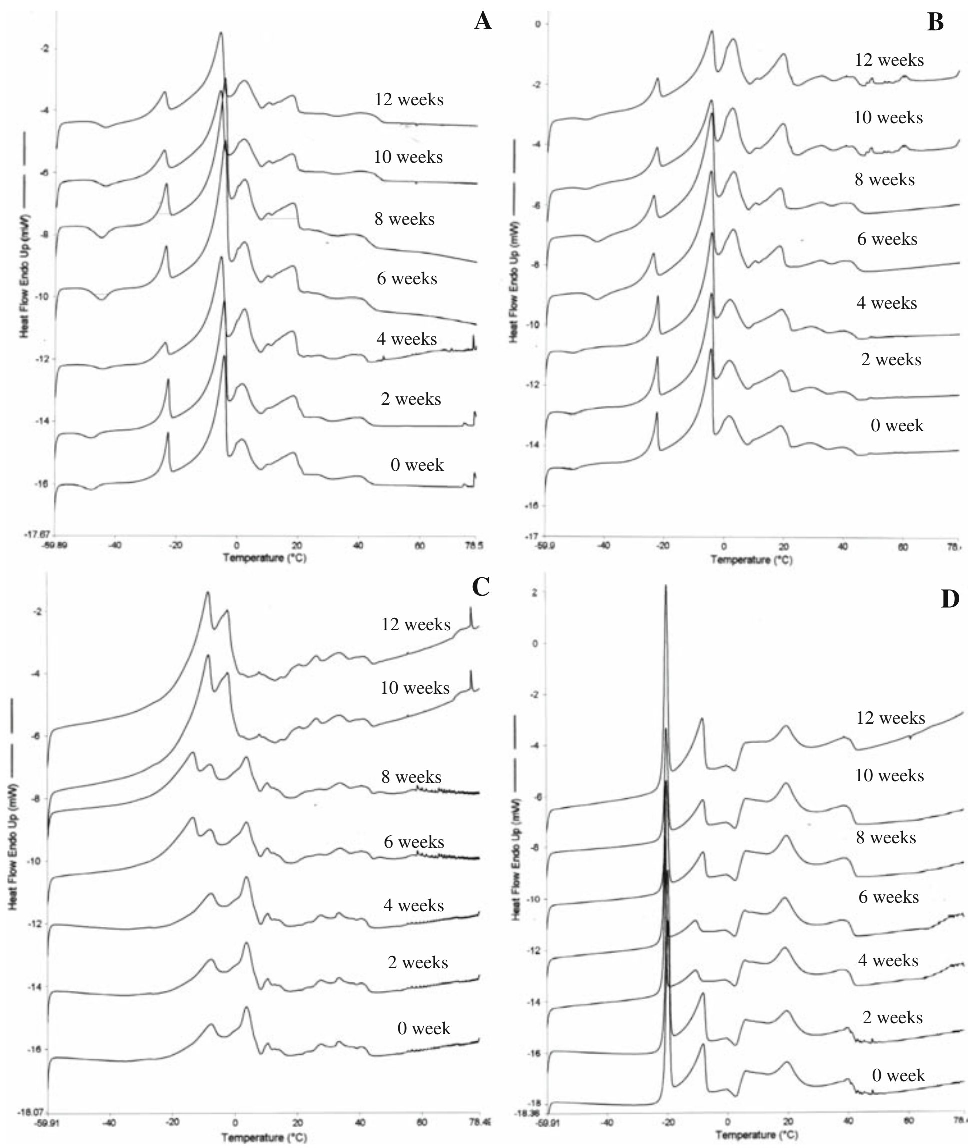

Fig. 3 DSC Melting curves of all different margarines throughout the 12 weeks storage period **a** Margarine DOS720, **b** Margarine DOS721, **c** Margarine DOS711 and **d** Commercial Margarine

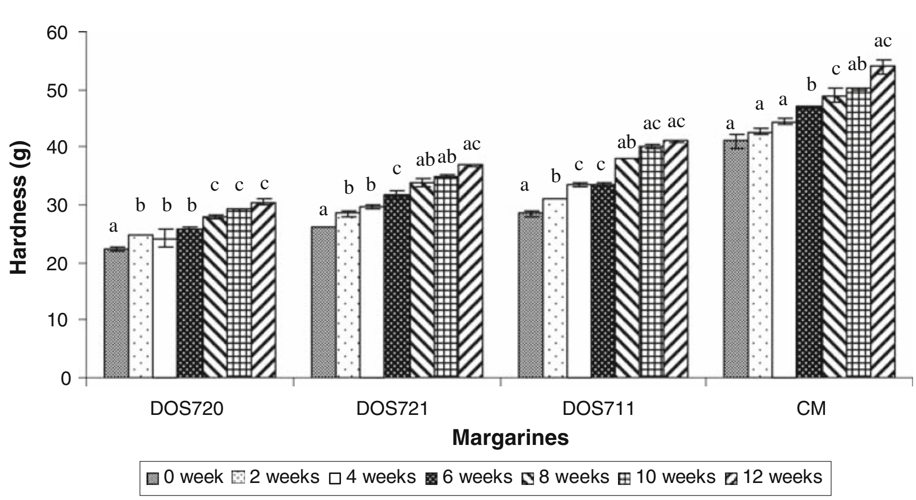

Fig. 4 Changes in hardness of PDG bakery margarines (Margarines DOS720, DOS721 and DOS711) and commercial margarine throughout the storage period of 12 weeks

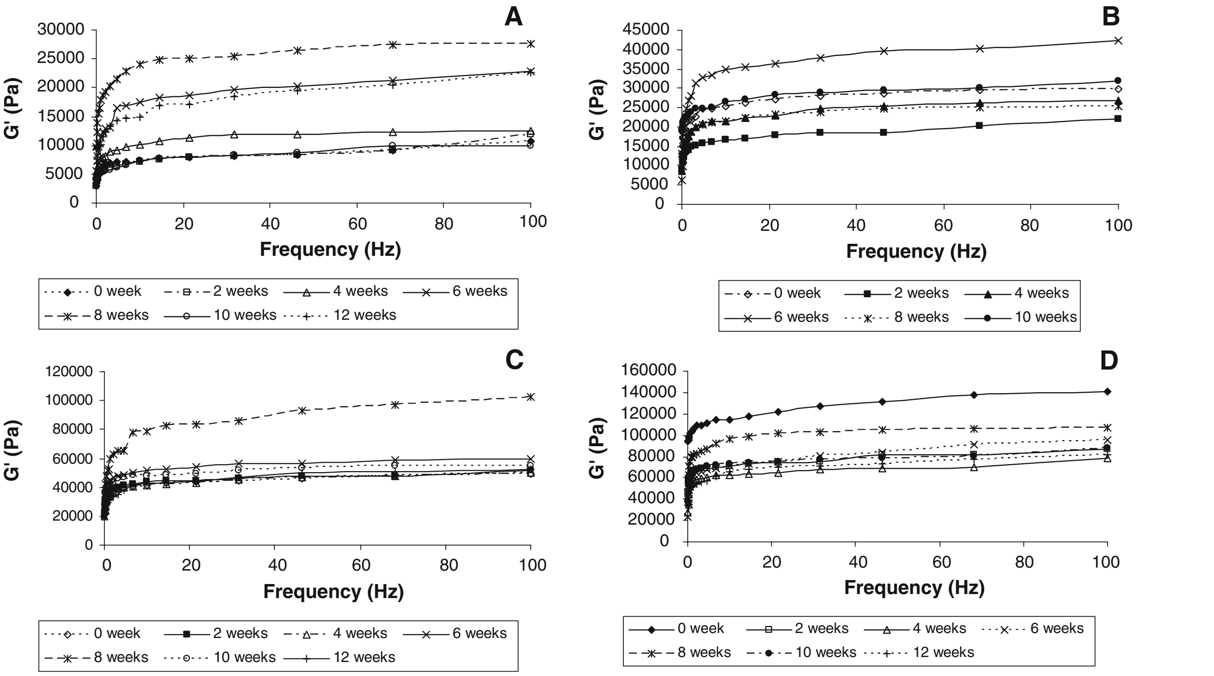

Fig. 5 Storage moduli (G') as a function of frequency (Hz) of all different margarines throughout the 12-week storage period **a** Margarine DOS720, **b** Margarine DOS721, **c** Margarine DOS711 and **d** Commercial Margarine

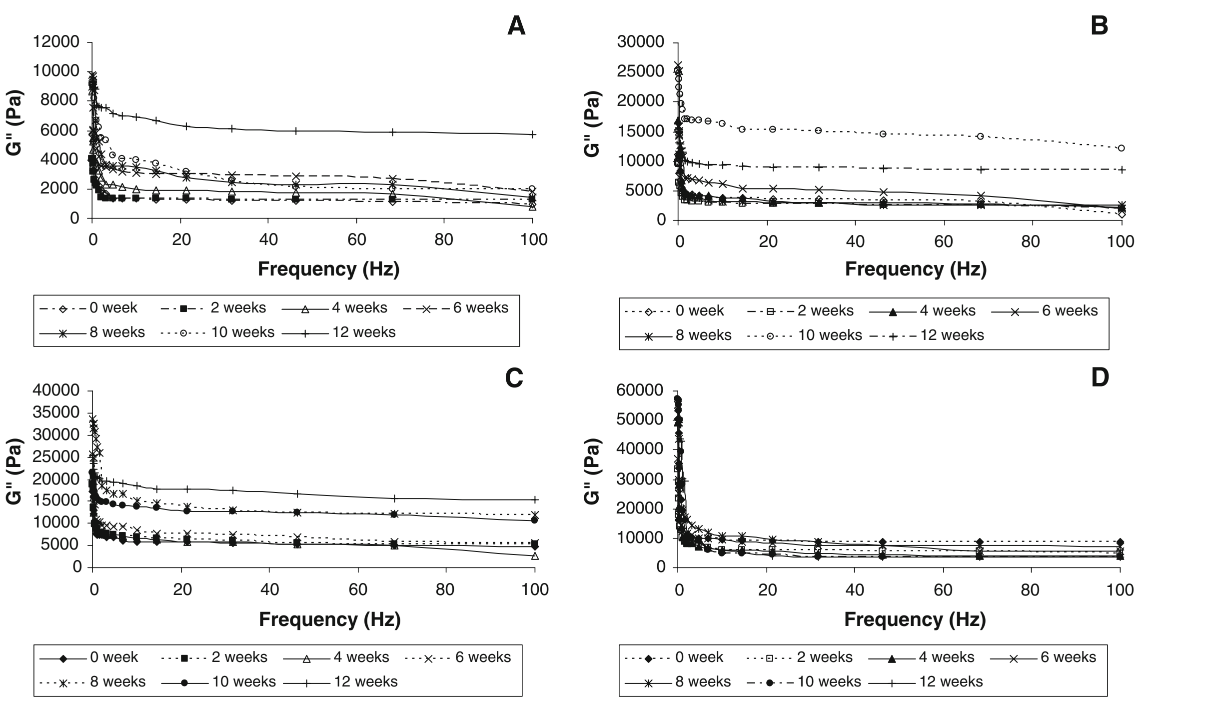

Fig. 6 Loss moduli (G'') as a function of frequency (Hz) of different margarines throughout the 12-week storage period **a** Margarine DOS720, **b** Margarine DOS721, **c** Margarine DOS711 and **d** Commercial Margarine

which shows that the values of tan delta were all below 1. High G', low G'' and tan delta values in margarines indicate that the fat crystal aggregates prevent translational movement; hence, the viscoelastic solid-like behavior [17].

A closer examination of the values of G', G'' and tan delta for all margarines during the storage period shows significant ($P < 0.05$) increments in the values of G' and decrements in the values of G'' throughout the 12 week

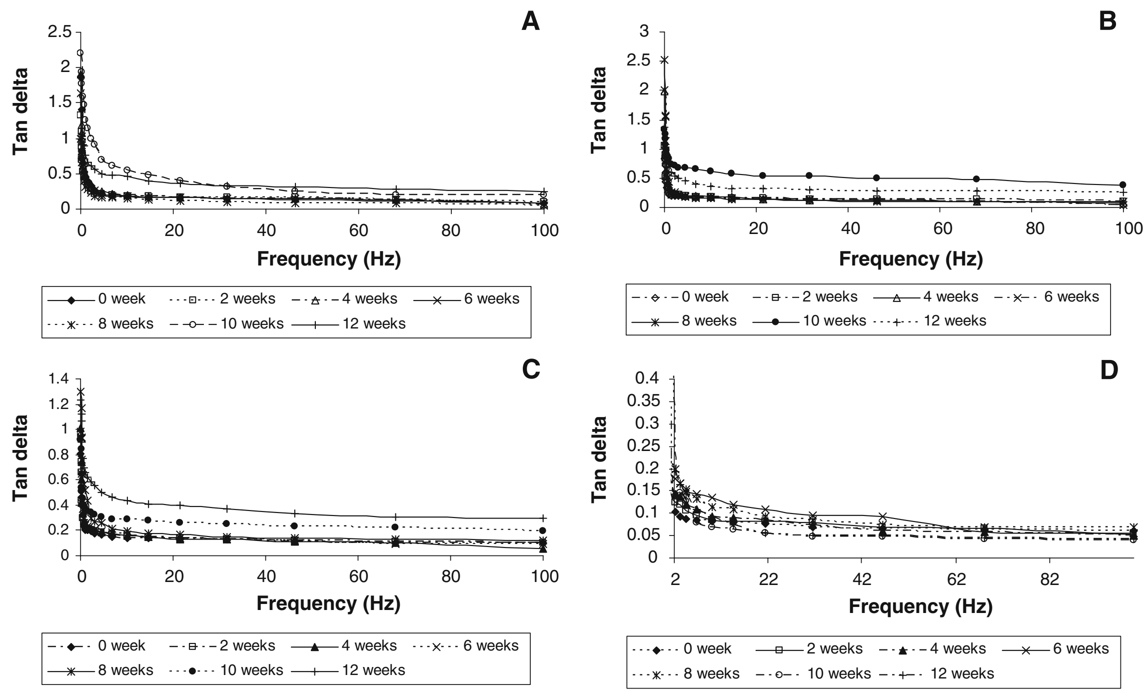

Fig. 7 Tan delta as a function of frequency (Hz) of different margarines throughout the 12-week storage period **a** Margarine DOS720, **b** Margarine DOS721, **c** Margarine DOS711 and **d** Commercial Margarine

storage period. This indicates all margarines had increased firmness at the end of the storage weeks which is in accordance with previously discussed findings. Higher degree of firmness in margarines at the end of the weeks of storage was mainly due to rearrangement of the fat crystals into a three-dimensional scaffolding network.

FTIR Analysis

Due to its small needle-like crystals which impart smooth texture and better organoleptic properties, β' crystals are desirable in margarines. Table 2 shows the subcell packing and polymorphic forms for all different margarines throughout the 12-week storage period. Up until 6 weeks of storage, all margarines had doublets in the rocking r(CH_2) region of 720 and 726 cm^{-1} in addition to a single band in the scissoring $\delta(CH_2)$ region of 1,470 cm^{-1}. These indicate that all the margarines had a mixture of β' and β crystals.

After 8 weeks of storage, CM only had a single band in the $\delta(CH_2)$ region of 1,470 cm^{-1}. This indicates that CM had undergone polymorphic transformation from a mixture of β' and β crystals to only β crystals. Polymorphic transformation from a mixture of β' and β crystals to only β crystals in CM had resulted in the formation of a firmer product. This is in accordance with previously discussed findings on increments in SMP, SFC and hardness of CM at the end of the 12-week storage period.

PDG bakery margarines with the exception of margarine DOS711 were able to retard polymorphic transformation

Table 2 Subcell packing and polymorphic forms of all different margarines throughout the storage period

	Subcell packing				Polymorphic form			
	DOS720	DOS721	DOS711	Commercial margarine	DOS720	DOS721	DOS711	Commercial margarine
0 week	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$\beta' + \beta$	$B' + \beta$	$\beta' + \beta$	$\beta' + \beta$
2 weeks	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$
4 weeks	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$
6 weeks	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$
8 weeks	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$T_{//}$	$\beta' + \beta$	$\beta' + \beta$	$\beta' + \beta$	β
10 weeks	$T_{//}$	$O_\perp + T_{//}$	$O_\perp + T_{//}$	$T_{//}$	$\beta' + \beta$	$\beta' + \beta$	β	β
12 weeks	$T_{//}$	$T_{//}$	$T_{//}$	$T_{//}$	β	β	β	β

for up to 10 weeks. After 12 weeks of storage, all the PDG bakery margarines also had only a single band in the $\delta(CH_2)$ region of 1,470 cm^{-1}; hence, only β crystals were present. Thus, once again PDG has shown to be able to retard undesirable polymorphic transformation. It is noteworthy that margarine DOS711 underwent polymorphic transformation slightly earlier than the other PDG bakery margarines. After 10 weeks of storage, margarine DOS711 had only β crystals. The earlier crystals transformation from β' to β form in margarine DOS711 may be due to the higher amount of PS in the margarine formulation. Higher amounts of palm stearin may have led to faster fat crystals agglomeration which not only interfered with the PDG ability to retard crystals transformation but also increased SMP, SFC and hardness of the products significantly.

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