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QUALITY SOFT TABLE MARGARINE WITH MINIMAL POST-CRYSTALLISATION THROUGH HIGH PRESSURE PIN-ROTOR UNIT

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ABSTRACT

Post-crystallisation is a hardening phenomenon in margarine and shortening after production. The choice of oils and fats, diacylglycerol (DAG) content and processing are several ways identified that could overcome post-crystallisation problem. There have been studies to understand this phenomenon, through the effects of emulsion temperatures, throughput speed, tube-cooler temperatures and pin-worker speed. Post-crystallisation in non-hydrogenated margarine during storage is significantly related to processing. Thus, minimising the post-hardening problem in palm-based soft margarine by studying the performance of high pressure working unit of margarine pilot plant will be the objective of the current work. A formulation containing palm oil as the major oil and a soft oil with a balanced ratio of 1:1:1 in saturated, monounsaturated and polyunsaturated fatty acids was processed in a margarine pilot plant. For evaluating the effects of high pressure coupled with pin-rotor, a high-speed pin-rotor and a double stage homogeniser were installed. The oils and fats and the finished products were analysed for their fatty acid composition, solid fat content, texture and hardness value upon storage. Post-crystallisation was greatly minimised in the soft margarine with the maximum usage of non-hydrogenated palm oil formulation at controlled storage temperatures of 5°C, 10°C, 15°C and 20°C.

Keywords: quality, palm oil, crystallisation, margarine, heat exchanger.

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INTRODUCTION

Oils and fats not only play a crucial part in human diet but has more important role in improving the palatability of foods. They are also the major ingredients of margarine. Due to that, palm oil (PO), being the most versatile oil is becoming an important raw material of choice for food manufacturers especially for producing margarine and shortening (De Clercq *et al*., 2012). Being naturally semi-solid and the commitment of oil palm growers to adopt the sustainable palm oil certifications such as the Malaysian Sustainable Palm Oil (MSPO) (Kushairi *et al*., 2017), now PO is the sustainable natural source of non-hydrogenated hard and semi-solid fats.

Oils and fats industry needs to continue and improve any technological problems. With regards to maintaining the natural source of fat, post-crystallisation has been a problem in margarine and shortening with high percentage of non-hydrogenated PO and palm stearin (DeMan and DeMan, 1994). Post-crystallisation is a hardening phenomenon in margarine and shortening after production. This problem has long been discussed by several researchers and some solutions have also been proposed. Controlling the diacylglycerol (DAG) content could be one of the ways to

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overcome the post-crystallisation problem, but may affect the melting properties of the final product. It was also suggested that high pressure pin-rotor be used which eventually could increase the rate of crystallisation.

Previous studies have shown that post-crystallisation in palm-based margarines and shortenings had been associated with their slower crystallisation behaviour as compared to the hydrogenated oils. Hence, in some cases, this lead to processing difficulties (Duns, 1985). This phenomenon increases the hardness of finished products several weeks after production. According to Timms (1984) and Duns (1985), the post-crystallisation might be due either to the transformation of *beta* prime to *beta* form of crystals or crystal networking after crystallisation. Blending of hydrogenated oil with other oils could reduce the transformation process by increasing the rate of crystallisation of the whole formulation (Moziar *et al.*, 1989) and stabilised the product consistency during storage (DeMan *et al.*, 1989). However, according to Yap *et al.* (1989), despite the slow crystallising behaviour of PO, the hydrogenated PO behaves better than other hydrogenated oils. PO was found to influence other oils with *beta* crystal property to crystallise excellently in *beta* prime form when blended together.

During processing, which mainly involves crystallisation, cooling conditions may not have reached the optimum temperature required for a thermodynamic equilibrium between the solid and the liquid phases to develop. This could result in recrystallisation. A good product during processing is not an exception. It could also experience recrystallisation especially in uncontrolled transportation and retail conditions leading to the development of new solid phases (Smith *et al*., 1994; Hishamudin *et al*., 2011). Crystal growth could also lead to the formation of solid bridges (sintering) between the narrow gaps of the crystal networks upon storage (Johansson *et al*., 1995) which could also contribute to the recrystallisation phenomenon.

It has been reported that post-crystallisation in a palm oil/sunflower oil soft margarine formulation could be minimised by optimising the processing conditions (Miskandar and Zaliha, 2014). Several other studies were conducted to tackle the post-crystallisation problem covering the areas of processing such as the effects of emulsion temperatures, throughput speed, tube-cooler temperatures and pin-worker speed. The study showed that varying the emulsion temperature in the mixing tank changed the solid fat content (SFC) and product temperature in the tank as well as in the tube-cooler and pin-worker, but the consistency of the product was not significantly changed during storage (Miskandar *et al*., 2002). According to Miskandar *et al.* (2002) although the pin-worker promoted crystallisation, the generated mechanical heat and endothermic heat of crystallisation caused some of the meta-stable crystals to melt. The mechanical action of the pin-worker also destroyed some of the crystal bonds creating an extremely large number of small crystals and increase the SFC. Throughput speed had been reported to have significant effects on the consistency and polymorphic transition of the product. Scraped-surface tube-cooler with a temperature at 20°C, on the other hand, showed an unexpected hardening of the margarine with the formation of *beta* crystal after the second week of storage (Miskandar *et al.*, 2002). This is in agreement with Chateris and Keogh (1991) who claimed that a scraped surface heat exchanger (SSHE) unit will provide two important treatments; heating or cooling treatment and shearing of the product.

The rotation speed of SSHE has an important role in determining the rate of crystallisation by facilitating contact between the fat and the cooling surface. According to Baccar and Abid (1999), this rate of contact could be related to the rotational velocity that created turbulent flow of the product on the cooling surface. The minimum speed that would create this turbulent was 8 rpm for a two-bladed (knife) SSHE. Fat crystallisation created by SSHE has an important effect on the final product. The rate of crystallisation especially on whether crystal formation should be allowed to occur on the surface of SSHE or after exiting the SSHE was studied by Qin *et al.* (2003). This is mainly due to the liberation of latent heat of crystallisation on the cooling surface of SSHE which also increased the heat transfer. Rao and Hartel (2006) studied the effects of varying SSHE temperatures on product quality. It was reported that the temperature differences between the inlet and the outlet in an SSHE could be achieved by inducing high throughput speed.

Zaliha *et al.* (2015) had quantified the phenomenon of post-crystallisation in their proposed mathematical model that predicts the degree of post-crystallisation upon storage at various temperatures. The formula had integrated the possible results of post-crystallisation in margarine using minimal parameters such as SFC, storage temperature and firmness to predict the final hardness as follows:

$$PHI = - b1(X1) + b2(X2) + b3(X3) + e \ldots\ldots\ldots\ldots (1)$$

where;

PHI - post-hardening index
b1-3 - regression estimates/coefficients
X1 - temperature (°C)
X2 - solid fat content (%)
X3 - firmness (g force)
e - error

The PHI developed clearly shows its dependency upon the process involved in the margarine production that contributes to the final product hardness. Since several studies involving processing had been conducted, while the effects of working the product under high pressure have not been studied by any researchers, the present study aimed to minimise the problem of post-crystallisation in a non-hydrogenated palm-based soft margarine by using a pilot scale margarine plant added with a high pressure pin-rotor unit.

MATERIALS AND METHODS

Materials

Refined, bleached and deodourised (RBD) palm stearin (POS) [iodine value (IV=35)], neutralised, bleached and deodourised (NBD) sunflower oil (SFO) and RBD palm olein (POO) (IV=58) were purchased from Mewah Oleo Sdn Bhd, Klang, Selangor, Malaysia. Mono and diacylglycerols, as emulsifier, were obtained from Danisco (M) Sdn Bhd, Pulau Pinang, Malaysia. Vacuum dried salt was purchased from a sundry shop, and water were obtained from regular water supply.

Methods

Formulation of soft table margarine. A series of formulations suitable for the soft table margarine was prepared based on their saturated (S), monounsaturated (M) and polyunsaturated (P) fatty acid compositions at the ratios of 2:1:1 - 1:1:1, as shown in *Table 1*. One of the formulations was selected for production in a pilot plant. The soft table margarine produced was used as samples in the study.

The ratio would provide a fair effect on the crystal formation and final product consistency as a result of the fatty acid composition during storage at various temperatures. *Table 1* shows the fatty acid composition of the blends containing POS, POO and SFO. The formulation with the ratio closest to 1:1:1 was selected as shown by the formulation number 8. Analysis on SFC, shows the sufficient SFC of the blend at temperatures ranging from 5°C to 40°C as shown in *Table 2*. Besides providing information on the spreadability of margarine formulation, SFC has another significant importance for a margarine producer. The amount of solid at a particular temperature could assist the producer in deciding the right temperature for the scrape surface exchanger (Miskandar and Noor Lida, 2011).

Margarine production. The margarines were produced using the usual table margarine process conditions as reported by Miskandar and Zaliha (2014). However, the addition of a high speed pin-rotor and double stage pressure homogeniser to the system was a process modification conducted for this study. The effects of pressure and working on the product during processing were achieved by varying the processing pressures from 3.45 - 32.40 bar and with high pin-rotor speeds of 500-2000 rpm as indicated in the response surface methodology (RSM) model (*Table 4*).

TABLE 1. FATTY ACID COMPOSITION OF THE SELECTED BLENDS

Formulations	Oils and fats			Fatty acids		
Number	POS	POO	SFO	Saturated	Monounsaturated	Polyunsaturated
1	0.05	0.59	0.35	37.5	32.5	30.0
2	0.12	0.52	0.37	38.3	31.2	30.4
3	0.03	0.57	0.41	35.2	31.8	33.0
4	0.13	0.47	0.40	37.6	30.4	32.0
5	0.08	0.48	0.44	35.0	30.2	34.8
6	0.13	0.46	0.42	37.0	30.0	33.0
7	0.09	0.54	0.37	37.6	31.5	30.9
8	0.07	0.50	0.43	35.3	30.5	34.2
9	0.11	0.48	0.41	36.8	30.4	32.8

Note: POS - refined, bleached and deodourised (RBD) palm stearin; POO - RBD palm olein; SFO - neutralised, bleached and deodourised sunflower oil.

TABLE 2. SOLID FAT CONTENT (%) OF SAMPLE 8 AT TEMPERATURES 5°C-40°C

	Temperature (°C)							
Sample code	5	10	15	20	25	30	35	40
Sample 8	30.5	24.7	18.5	12.1	8.5	7.0	4.7	3.0

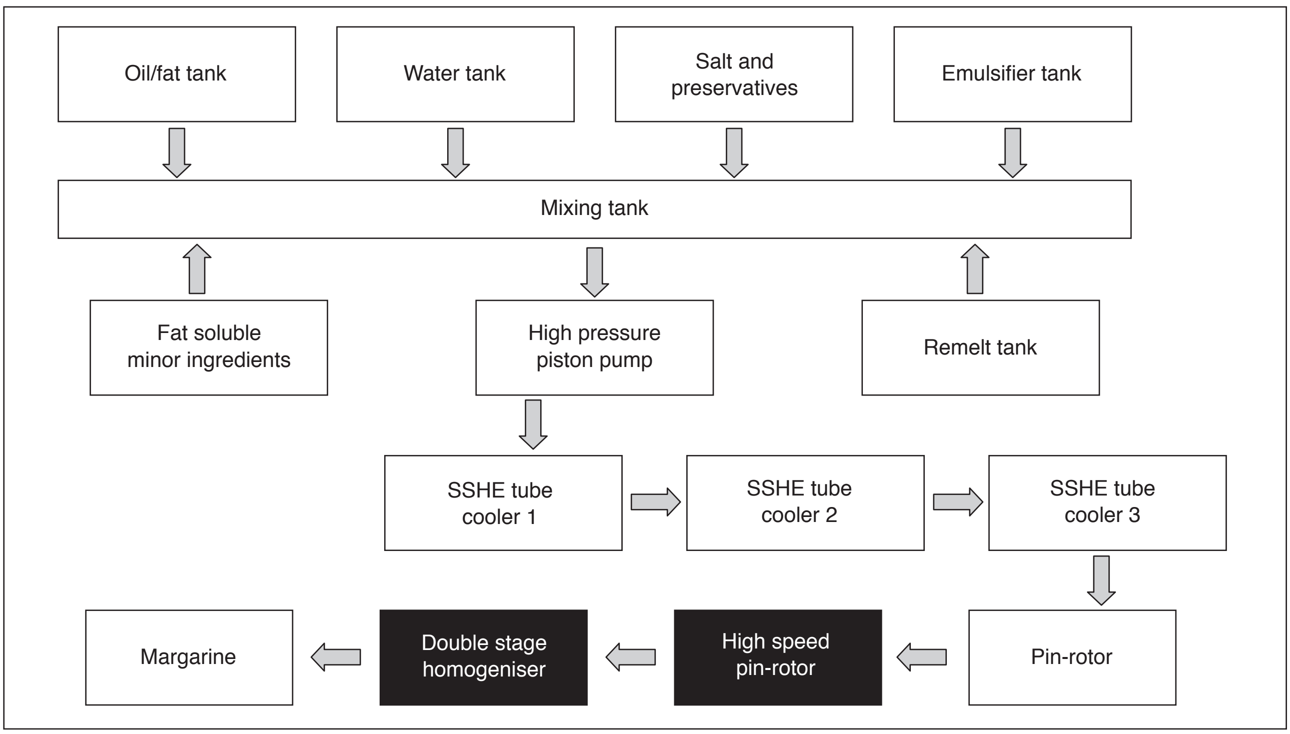
Note: Sample 8 - selected formulation from *Table 1*.

The experimental design was based on the customised soft margarine production conducted in the Malayasia Palm Oil Board (MPOB). *Figure 1* shows the overall flow chart of margarine production, starting from the initial preparation to the filling stages. The following combination was used: 81.7% fat phase, 0.3% emulsifier, 16% water, and 2.0% salt. Oils and fats (sample 8) were melted in a Memmert drying oven (854 UL 80, Schwabach, Germany) at 65°C, then weighed accordingly for 50 kg production batches.

The emulsifier was first melted in a small portion of the fat blend at a ratio of 1:4 before adding it to the fat phase. The water phase at 28°C was then added slowly to the oil phase with agitation to form a stable emulsion (Anderson and Williams, 1965; Faur, 1996; Hui, 1996; Miskandar *et al*., 2002; Goli *et al*., 2009). The emulsion temperature was maintained at 55°C and held for 15 min in the mixing tank prior to processing in a perfector pilot plant (Gerstenberg and Agger, Copenhagen, Denmark) at MPOB. The tube cooler SSHE has a volume of 900 ml and scraped cooling surface area of 0.063 m^2. Two tube coolers, SSHE1 and SSHE2, were set at standard temperatures for the whole study at 10°C and 20°C, respectively. The pin worker (pin-rotor), with a volume of 3 litres, was set after the SSHE 2. The emulsion was pumped into the SSHE 1 (at throughput rate of 45-65 kg hr^{-1}) where it was rapidly cooled. The rotation speeds of SSHE1, SSHE2 and pin-rotor were 1000, 1000 and 100 rpm, respectively. The margarine then entered the high speed pin-rotor (HSPR) operating at rotation speeds of 514 -1786 rpm. The margarine exited the processing system via a double stage homogeniser (DSH), operating at pressures of 0 – 32.52 bar. The margarine samples collected at the end of the processing line were filled in a total of 75 400 ml, bowl shaped plastic containers and tempered at 5°C for 24 hr before placing them in 5°C, 10°C, 15°C and 20°C incubators for a minimum of 25 days.

Experimental design. Sample 8 (as in *Table 1*) was processed in nine different processing parameters. *Table 3* shows the processing model of this study, such as processing in high HSPR and high DSH pressure, is coded as HSHP (*Table 3*), while processing in low HSPR speed and medium DSH pressure is coded as LSMP (*Table 3*). These processing models are translated in the experimental design as indicated by the critical factors and responses as shown in *Table 4*. Three critical outputs of the products namely hardness, (g cm^{-2}), hardening rate and SFC were tabulated and used as the response output using RSM (Saberi *et al*., 2011; Miskandar and Zaliha, 2014).

Analyses. Fatty acid composition (FAC), slip melting point (SMP), SFC and isothermal solid fat content (ISFC) were determined following MPOB Test Methods (MPOB, 2005). The consistency of the margarine was determined by measuring the



Note: SSHE - scrapped surface heat exchanger.

Figure 1. Flow chart of margarine processing, showing the position of the high speed pin-motor and double stage homogeniser (modified from Miskandar and Zaliha, 2014).

TABLE 3. PROCESSING MODEL

	High pressure (> 27.5 bar)	Medium pressure (15.5 bar)	Low pressure (0-3.45 bar)
High speed (>1600 rpm)	HSHP	HSMP	HSLP
Medium speed (1150 rpm)	MSHP	MSMP	MSLP
Low speed (<700 rpm)	LSHP	LSMP	LSLP

Note: HSHP - high high speed pin-rotor (HSPR) speed and high double stage homogeniser (DSH) pressure.
HSMP - high HSPR speed and medium DSH pressure.
HSLP - high HSPR speed and low DSH pressure.
MSHP - medium HSPR speed and high DSH pressure.
MSMP - medium HSPR speed and medium DSH pressure.
MSLP - medium HSPR speed and low DSH pressure.
LSHP - low HSPR speed and high DSH pressure.
LSMP - low HSPR speed and medium DSH pressure.
LSLP - low HSPR speed and low DSH pressure.

Statistical analyses. Optimisation of the processing condition was done by RSM statistical package in the Design Expert 6. Model of 11 process conditions including three control conditions with ANOVA at 95% confidence level on samples of three replications was chosen. A few experiments were then selected to demonstrate the variable effects.

In meeting the objectives of the project, a simple equation by Microsoft Excel below was used.

$Y = MX+H$

where,

Y - predicted hardness of margarine

M - weekly hardening rate, g cm^{-2} wk^{-1}

X - number of weeks (forecasted)

H - initial hardness, g cm^{-2}

The line of best fit derived from the regression analysis of hardness as a function of time was used to determine the extent of post-crystallisation in the margarine based on true margarine processing in a pilot scale plant. The slope, *M*, represents the weekly hardening rate (g cm^{-2} wk^{-1}) of the margarine during storage. A smaller slope value is preferred, as it indicates lower hardness increment weekly. The analyses were also conducted on a complete margarine formulation and storage study was conducted at specific temperatures.

TABLE 4. EXPERIMENTAL DESIGN SHOWING THE PRODUCT RESPONSES [hardness (g cm^{-2}), hardening rate (g cm^{-2} per week), and solid fat content] AT THE END OF 25 DAYS STORAGE AT 5°C

Std	Critical factors			Responses		
	Code	HSPR speed (rpm)	DSH pressure (bar)	Hardness (g cm^{-2})	Hardening rate (g cm^{-2} per week)	SFC (%)
1	1073	700	3.45	950	21	16
2	1074	1 600	3.45	900	20	15
3	1350	700	27.56	1 400	106	14
4	1076	1 600	27.56	900	83	22
5	1077	524	15.50	1 200	130	17
6	1078	1 786	15.50	1 050	28	18
7	1079	1 150	0	820	19	20
8	1080	1 150	32.52	950	34	21
9	1081	1 150	15.50	1 300	59	18
10	1082	1 150	15.50	1 200	42	19
11	1084	1 150	15.50	800	-4.1	19

Note: 1073 (LSLP), 1074 (HSLP), 1350 (LSHP), 1076 (HSHP), 1077 (LSMP), 1078 (HSMP), 1079 (MSLP), 1080 (MSHP), 1081 (MSMP), 1082 (MSMP), 1083 (MSMP), 1084 (MSMP), 1085 (MSMP). HSPR - high speed pin-motor. DSH - double stage homogeniser.

depth that a needle cone with a specific angle and weight penetrated the margarine from its surface for a given time and at a specific temperature. The cone was dropped freely by the force of gravity. The penetration value is referred to as hardness, g cm^{-2} (Haighton, 1976; DeMan *et al*., 1989) using a cone penetrometer (Stanhope-Seta, Surrey, England) with 40° cone, weight of 79.03 g with penetration time of 5 s. The calculation was following the formula described by Haighton (1976), hardness, $H = KW/P^{1.6}$, where K = 5840, W = 79.03 + added weight, and P = mean of penetration readings. Six readings were taken from each sample daily from different sub-samples. Microscopic examination for crystal distribution was performed as described by Miskandar *et al*. (2004). Texture was also analysed to assess the quality of the margarine.

RESULTS AND DISCUSSION

The Effects of High HSPR Speed (rpm) and DSH Pressure (bar) on the Hardness (g cm^{-2}) of Margarine as a Function of Storage (day)

The hardness of soft margarine during storage was affected by the DSH pressure applied during processing. When the soft margarine was processed in a high HSPR speed, and with high DSH pressure applied opposing the flow of the product exiting the perfector unit, the product showed a significant consistency with high hardness of 1200 g cm^{-2} at Day 25 (*Figure 2a,* HSHP). When the pressure was reduced, the hardness of the margarine reduced significantly as shown in *Figures 2b* and *2c* (HSMP and HSLP).

A simple rule applies in discussing the above result. As high DSH pressure was applied opposing the exit of the product, residential time of the margarine was increased in the SSHE. More crystallisation had taken place and subsequently increased the consistency (Miskandar and Zaliha, 2014). As the margarine flowed passing through the HSPR, crystal aggregates would be broken into smaller aggregates and reduced the spaces in between them (Miskandar *et al.,* 2002). Crystal aggregates would become more compact and caused the consistency to increase.

Generally, the margarine had high hardness values as measured by yield value (g cm^{-2}) at lower storage temperatures of 5°C and 10°C. At higher storage temperatures of 15°C and 20°C, especially at 20°C, the hardness values were significantly lower than at other storage temperatures. This is mainly due to the SFC of the margarine that is significantly low at 20°C and above as shown in *Table 2.*

The hardness values of the margarine samples HSHP, HSMP and HSLP at 5°C were significantly different for the first 10 days. The values of the products range from 500 to 900 g cm^{-2}. Sample HSHP increased from 500 g cm^{-2} on the 1^{st} day to 900 g cm^{-2} on the 10^{th} day and increased significantly to 1400 g cm^{-2} at the weekly increment rate of 82 g cm^{-2} (*Figure 2).* Thereafter, the hardness reduced again to 1300 g cm^{-2}. On the other hand, HSMP showed a lower weekly increment rate of 28 g cm^{-2} , while HSLP was very similar to HSMP, at 20 g cm^{-2} . Similarly, the trend was somewhat the same for 10°C, but was lower for HSMP and HSLP.

Samples HSMP and HSLP, showed insignificant weekly hardness increment of 850 and 768 g cm^{-2} on the first day to 855 and 872 g cm^{-2} on Day 25, respectively, except for Day 10 in sample HSMP (1200 g cm^{-2}). There were no significant changes in hardnesss during storage at 15°C and 20°C for samples HSMP (29 and 4 g cm^{-2} weekly, respectively) and HSLP (-8 and -5 g cm^{-2}, respectively).

Since 5°C posses as a critical storage temperature for palm-based soft margarine, producing a product that is spreadable and stable for a long storage time at this temperature is desirable. A standard process condition with high HSPR and high DSH pressure has not been able to produce a consistent and spreadable product at 5°C, as shown by sample HSHP with a weekly hardness increment rate of 82.5 g cm^{-2}. On the other hand, samples HSMP and HSLP were leading a better lower, weekly hardness increment rate during storage at 5°C. The weekly hardness increment rate for HSMP and HSLP were 28 and 19.9 g cm^{-2}, respectively. The trends of the three process parameters suggested that as the DSH pressure is reduced, the weekly hardness increament rate is also lowered. The result might also indicate that margarine with good stability could be produced using a lower DSH pressure.

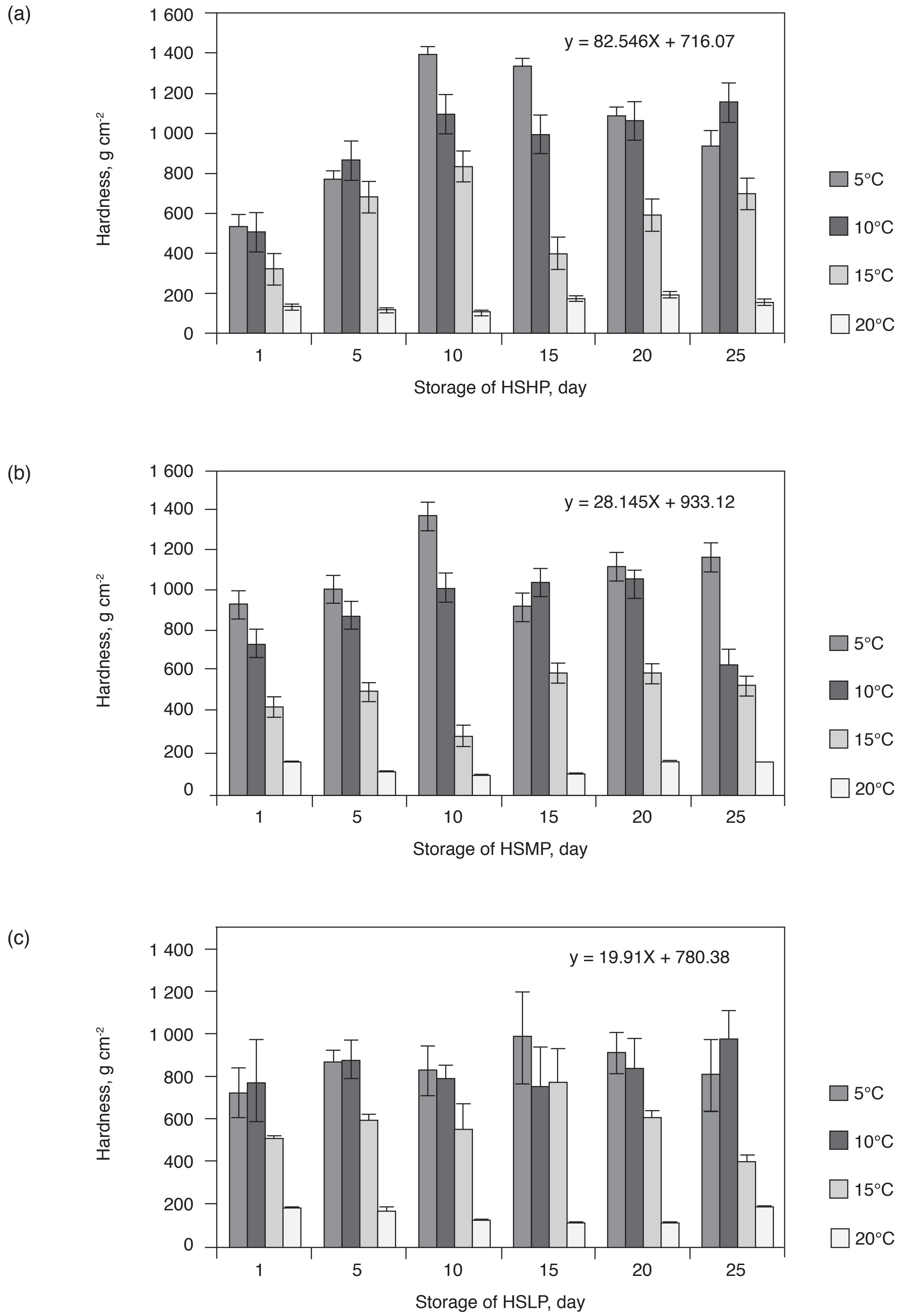
The Effects of Medium HSPR Speed (rpm) and DSH Pressure (bar) on the Hardness (g cm^{-2}) of Margarine as a Function of Storage (day).

Processing the margarine using medium HSPR speed, and various DSH pressures had resulted in interesting and useful data. A general observation on the effects of three different DSH pressures as a function of medium HSPR speed showed a reduction in hardness as the DSH pressure was reduced from 470 rpm to 0 rpm. At 5°C, the margarines had hardness values of < 1000 g cm^{-2}, indicating that the margarines were generally soft, although several parameters indicated samples that were significantly hard, >1000 g cm^{-2}. Storage of samples MSHP and MSLP at this temperature, had resulted in the occurrence of post-crystallisation at Day 10. Sample MSHP exhibited the worst amongst the others (*Figure 3*). Despite that, the consistency of the margarine sample, MSLP, was generally <1000 g cm^{-2}, at a weekly hardening rate of 19 g cm^{-2} (Y=19X+780) and this provided a lower post-hardening effect.

The samples, besides generally being soft, parameters MSHP and MSMP produced samples with >1000 g cm^{-2}, that were hard and consistent, at weekly hardening rates of 34 g cm^{-2} (Y=34X+956) and 59 g cm^{-2} (Y=59X+820), respectively. Overall, we could conclude that medium speed process conditions does not produce homogenous margarine at 5°C.

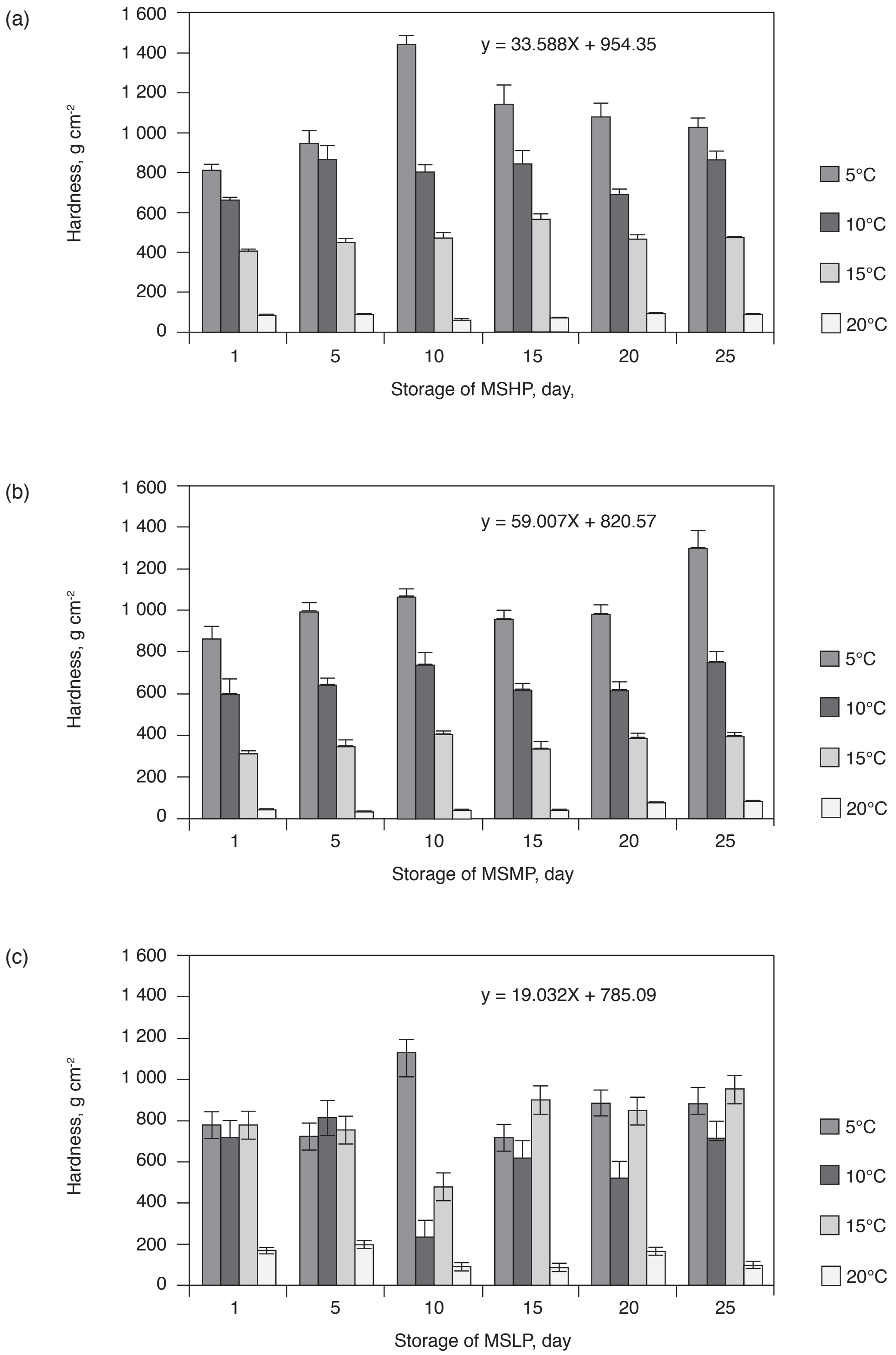
The Effects of Low HSPR Speed (rpm) and DSH Pressure (bar) on the Hardness (g cm^{-2}) of Margarine as a Function of Storage (day)

When the margarine was processed at low speed HSPR system at various DSH pressure conditions, from 27.56 to 0 bar and at various storage temperatures, the margarine had significantly high hardness for samples LSHP and LSMP at 5°C, as shown in *Figure 4*. The rate of hardness increment was 45 g cm^{-2} weekly for sample LSHP (Y = 45X+915), while sample LSMP showed an increase in hardening rate of 130 g cm^{-2} weekly (Y=130X+661). Changes in product hardnesss of samples LSHP and LSMP during storage at 15°C and 20°C, however, were not significant. The rate of weekly hardness increment for sample LSHP was 34 g cm^{-2} (Y=34X+464) at 15°C



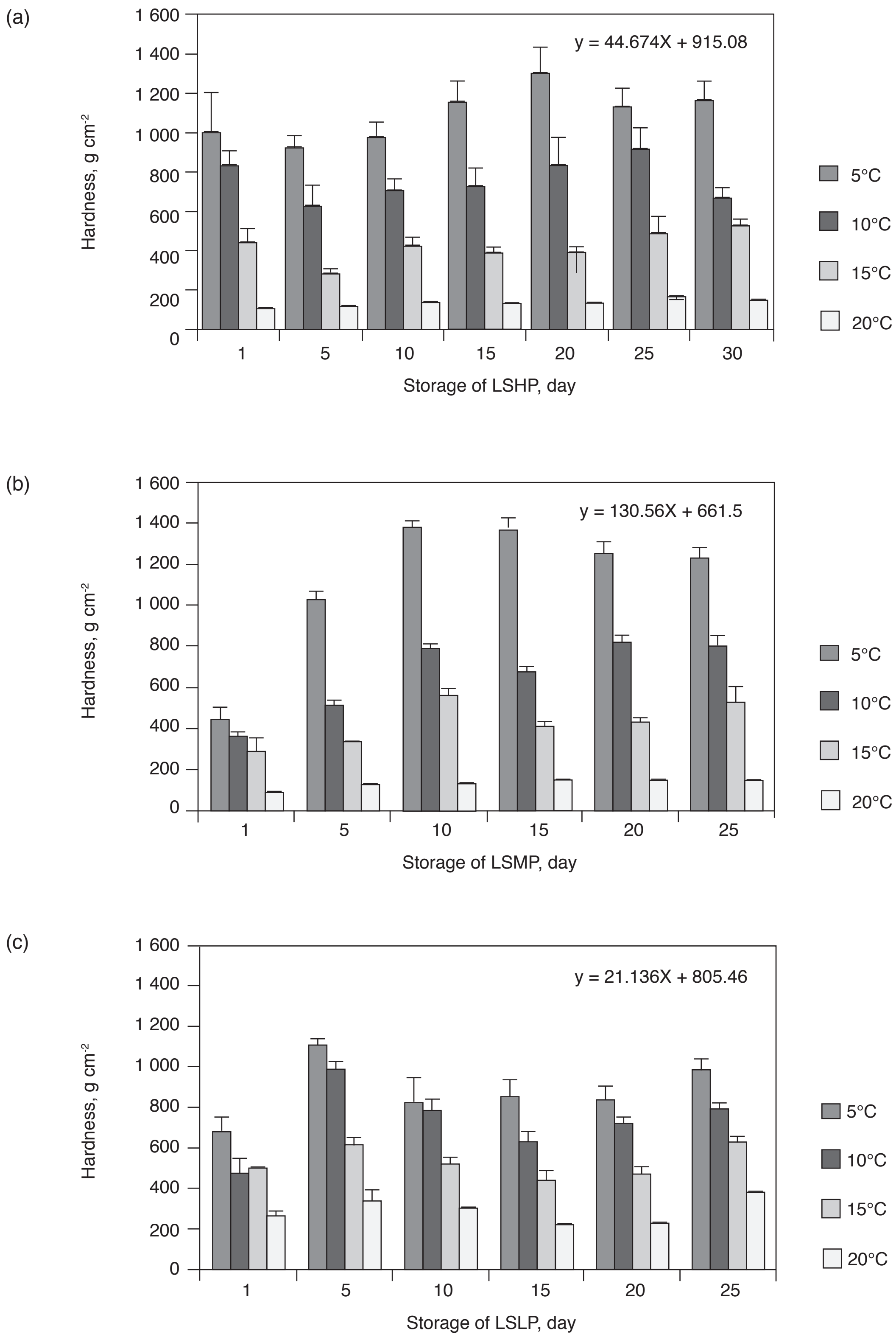
Note: HSPR - high speed pin-rotor.
DSH - double stage homogeniser.
HSHP - high HSPR speed and high DSH pressure.
HSMP - high HSPR speed and medium DSH pressure.
HSLP - high HSPR speed and low DSH pressure.

Figure 2. The effects of high HSPR (rpm) and (a) high (HSHP), (b) medium (HSMP), (c) low (HSLP) DSH pressures (bar) on the hardness (g cm^{-2}) of margarine as a function of storage (day).



Note: HSPR - high speed pin-rotor.
DSH - double stage homogeniser.
MSHP - medium HSPR speed and high DSH pressure.
MSMP - medium HSPR speed and medium DSH pressure.
MSLP - medium HSPR speed and low DSH pressure.

Figure 3. The effects of medium HSPR speed (rpm) and (a) high (MSHP), (b) medium (MSMP), (c) low (MSLP) DSH pressures (bar) on the hardness (g cm^{-2}) of margarine as a function of storage (day).



Note: HSPR - high speed pin-rotor.
DSH - double stage homogeniser
LSHP - low HSPR speed and high DSH pressure.
LSMP - low HSPR speed and medium DSH pressure.
LSLP - low HSPR speed and low DSH pressure.

Figure 4. The effects of low HSPR speed (rpm) and (a) high (LSHP), (b) medium (LSMP), (c) low (LSLP) DSH pressures (bar) on the hardness (g cm^{-2}) of margarine as a function of storage (day).

and 13 g cm^{-2} (Y=13X+96) at 20°C, while for sample LSMP it was 38 g cm^{-2} (Y=38X+290) at 15°C and 9 g cm^{-2} (Y=9X+96.8) at 20°C. Samples were stable and with good consistency, although the products exhibited soft texture. Sample LSHP was in the range of 1000 - 1300 g cm^{-2}, however it was still spreadable at 25°C or home temperature, and considered as still good.

In a high pressure operation, crystallisation is enhanced due to the prolonged residential time, causing efficient supercooling resulting in the increase of margarine consistency or hardness. At low HSPR speed, agglomerated crystal breakage will be minimised. Crystal packing is not easily formed during storage, thus product hardening usually does not occur (Miskandar and Zaliha, 2014).

Except for sample LSMP, sample LSHP had shown an insignificant increment in hardness during storage at 5°C, indicating that the product had reached in equilibrium crystal development during processing. This can be observed from the SFC development during storage, as shown in *Figure 5*. SFC development during storage as measured isothermally, shows consistent straight lines for all temperatures beginning the fifth day of storage.This indicates that crystal development had reached its equilibrium in all storage temperatures as early as the fifth day of storage. The result could conclude that this particular process condition could complete the crystallisation activities during processing with no significant changes at a controlled temperature condition.

Sample LSLP had shown a slight difference in its crystal development especially at 5°C of storage. The product was generally soft with hardness of < 1000 g cm^{-2}, except for Day 5. The hardness rate of this parameter was 21 g cm^{-2} weekly. At this rate, it could be predicted that the product would reach a hardness of 1355 g cm^{-2} after six months of storage at 5°C. At this hardness, generally the product would still exhibit good texture but would have lost most of its spreadablity quality and hardly deform at end-user temperature for serving.

In a low pressure operation, residential time was shortened, while crystallisation rate was increased. According to Rao and Hartel (2006), crystallisation at the SSHE is dependent on the temperature of the product throughput. In relation to this study, the low pressure had created a situation of additional crystal development rate from the standard process operation. Furthermore, a low HSPR speed, crystal agglomerates developed during the standard process operation was not significantly affected. There was minimal breakage of crystal agglomerates, which may have increased crystal compactness.

In terms of crystallisation activities, *Figure* 6 shows that isothermal SFC of the margarine started with lower SFC than LSHP at Day 1, especially at 5°C, while at all temperatures, they had a similar trend with hardness. There were no sigficant differences in the SFC during the fifth and 25th day of storage. This crystal development indicates that processing was efficient. The process condition was able to provide sufficient crystallisation requirement that allows fats and oils to crystallise completely as it exits the filling station. As this happens, fat recrystallisation process minimally occurs. Crystal developments have reached their equilibrium during the second week of storage, which is a normal phenomenon for non-hydrogenated fat-based margarine. The development of SFC isothermally at various temperatures as shown in *Figure 5* supported the theory that crystal development has reached equilibrium at all temperatures.

General observations on the integrated effects of pin-rotor speed and pressure applied on the product

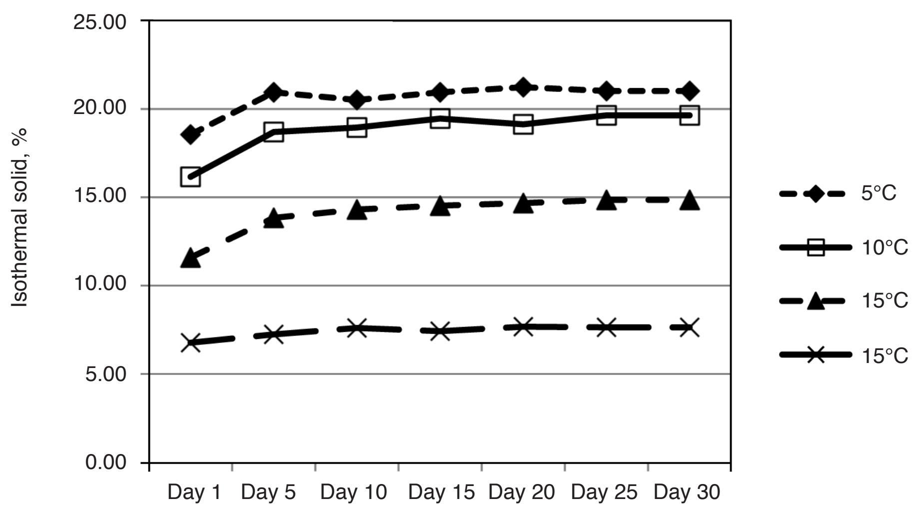


Figure 5. Solid fat content measured isothermally for samples processed in sample low high speed pin-rotor (LSHP) and high double stage homogeniser (DSH) pressure condition.

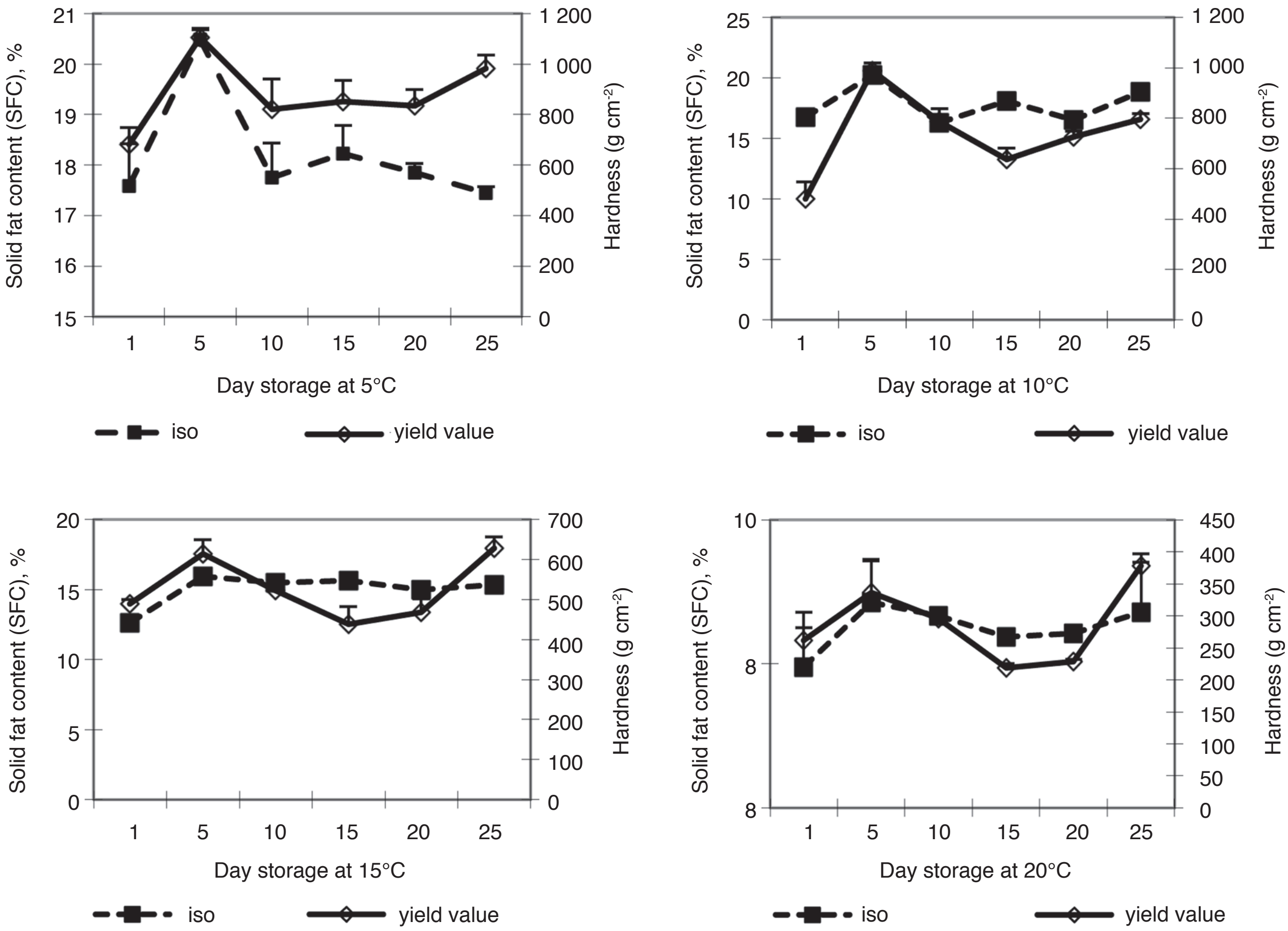


Figure 6. The trend of the isothermal solid of sample low high speed pin-rotor (HSPR) and low double stage homogeniser (DSH) pressure (LSLP) as compared to the trend of its hardness in the same storage temperatures.

exit has developed a trend as a guide. At low HSPR speed, the rate of weekly hardness increment of the samples at 5°C will increase, with the increase of pressure. However, there was no effect on the samples at 10°C, 15°C and 20°C.

Prediction of Margarine Quality Using RSM

Process conditions, particularly the temperature of the SSHE, flow rate of emulsion entering the SSHE unit and the rotation speed of the pin-rotor could affect product quality. According to Baccar and Abid (1999), rotational speed of a two-bladed SSHE requires a minimun of 8 rpm to create a turbulent emulsion in the SSHE, while the temperature differences between the inlet and the outlet in a SSHE depend on heat transfer and throughput speed (Rao and Hartel, 2006). Studying the effects of these parameters is thus essential and has been completed in the previous works. The current study focussed on solving the phenomenon of post-crystallisation due to the maximum usage of palm oil in the formulation. An additional processing aid, HSPR which can hold high pressure and increase the residential time of the margarine in SSHE was applied. The parameters that were computed from the RSM are shown in *Table 4*. *Table 4* shows the quality of soft margarine model coded under the column 'code', as indicated by hardness (g cm^{-2}) during storage of 25 days at 5°C. From the information obtained, weekly hardening rate was established. As hardening of margarine due to storage is also a reflectance of the degree of post-crystallisation, the parameters shown could establish the processing conditions to be used, when a stable margarine is to be produced by a manufacturer.

The data in *Table 4* was computed using the RSM software to generate a prediction of the effects of each parameter on the quality of the margarine. This is an established way of predicting the outputs of a process in a very cost effective and time saving way. The graph in *Figure 7* shows that when the DSH pressure is increased from 3.45-32.52 bar, the HSPR speed should be reduced from 1150 to 813 rpm, to obtain soft margarine with hardness values of 968-1106 g cm^{-2}. Thus, to achive a product with a desired hardness of 1244.17 g cm^{-2}, the operating HSPR speed should be set to 813 rpm, while the DSH pressure is set at 17.98 bar.

The graph in Hardness, indicates the hardening rate of the margarine every week during storage. Hardening reflects poor margarine quality that leads to difficulties in spreading on bread, brittleness and oil separation. A soft margarine needs to be processed using the right HSPR speed and DSH pressure during production. The graph indicates that for the margarine to have reduced weekly hardening rate, the DSH pressure should be reduced while the HSPR speed is increased. Thus, when the HSPR speed is 895 rpm and the DSH pressure applied is 3.45 bar, the hardening rate is <24.794 g cm^{-2} per week.

The computed suggested process conditions for producing soft margarine with predicted hardness, weekly hardening rate and SFC is shown in *Table 5*. It is predicted that when the HSPR speed is 1155 rpm and the DSH pressure applied is 3.45 bar, the hardness of the margarine will be 963 g cm^{-2} and the hardening rate is 27 g cm^{-2} per week.

Confirmatory Test

A confirmatory test run using formulation 8 in *Table 1*, and processed according to the process flow chart as in *Figure 1* was conducted, while incorporating the suggested HSPR speed and DSH pressure as in *Table 5*. The complete process conditions are shown in *Table 6*. The product was tempered at 5°C for 24 hr and storage studies were determined for 40 days.

Hardness developments of the margarines produced in HSPR of 1155 rpm and DSH pressure of 3.45, 6.89 and 10.34 bar, during a 40-day storage at 5°C are shown in *Figure 8*, while the texture of the margarines during the 5th and 30th day of storage are shown in *Figures 9, 10, 11* and *12*. *Figure 8* shows that the hardness of the margarine processed at 3.45 bar DSH pressure during the 40th day analysis at storage of 5°C was 1002 g cm^{-2}. The result is not significantly different from the predicted value of 900 g cm^{-2}. The hardening rate of the margarine using this condition is 20.4 g cm^{-2} per week. This is very close to the predicted value of 22 gcm^{-2} per week. The margarine has good texture and spreadability as shown by the smooth line curves as shown in *Figures 9* and *10* with spreadability values of 3123 g s^{-1} on Day 5 and 3668 g s^{-1} on Day 30, respectively.

The margarines produced in a 10.34 bar DSH pressure had higher hardness values as compared to 3.45 bar DSH. The observation is in compliance with the discussion above, while discussing on the effects of high HSPR speed and DSH pressure on the hardness of margarine as a function of storage. The texture of the margarines produced in 10.34 bar DSH is shown in *Figures 11* and *12*. The product was not homogeneous due to the formation of lumps of crystals as indicated by the sharp curve lines. At higher temperatures of 10 (*Figure 13*) and 15°C (*Figure 14*), the average product hardness was lower than 1000 g cm^{-2} during the 40 days of storage for all products produced under various DSH pressures.

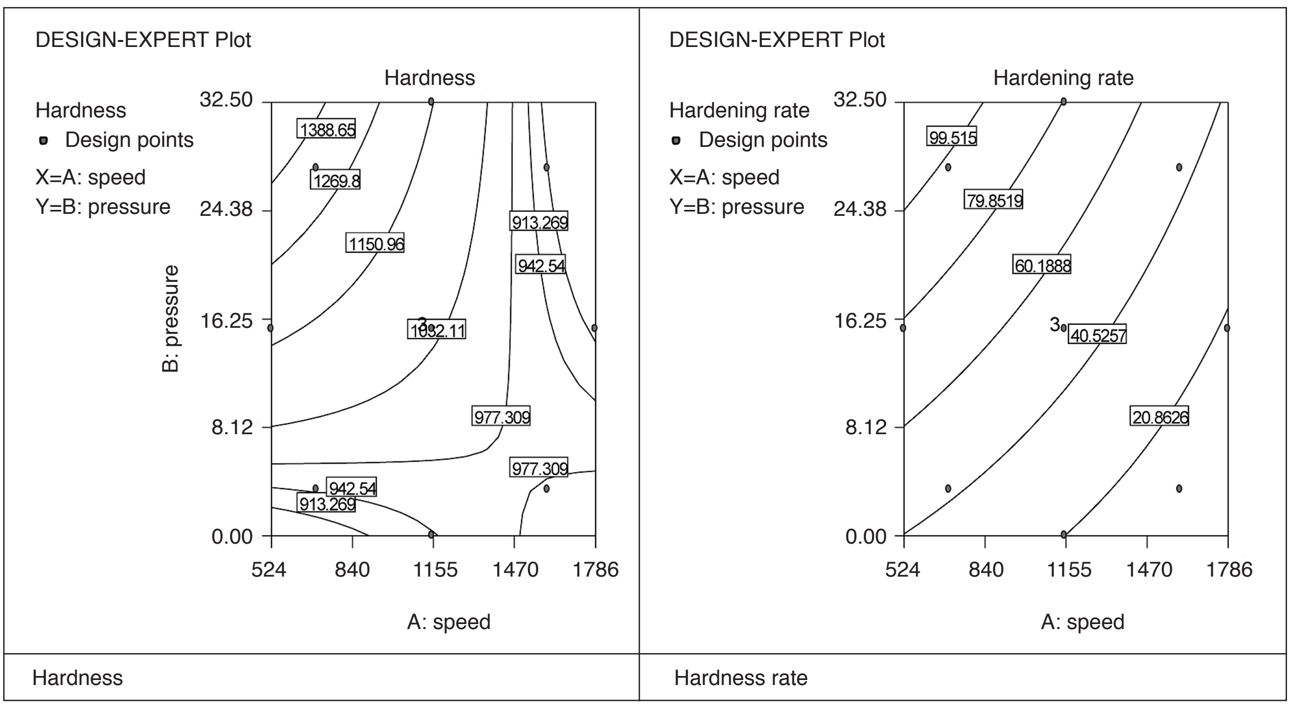


Figure 7. The effects of double stage homogeniser (DSH) pressure as a function of speed in the prediction of hardness (hardness) and hardening rate (hardening rate) of margarine.

TABLE 5. SUGGESTED CRITICAL PROCESS PARAMETERS THAT INCLUDE HIGH HSPR SPEED AND DSH PRESSURE AND THEIR PREDICTED RESULTS OF THE MARGARINE (sample 8)

Factor	Name	Level	Low level	High level	Std. dev.	Prediction
A	HSPR speed	1 155	524	1 786	0	-
B	DSH pressure	3.45	0	32.50	0	-
Hardness	-	-	-	-	-	963
hardening rate	-	-	-	-	-	27
SFC	-	-	-	-	-	17

Note: HSPR - high speed pin-motor.
DSH - double stage homogeniser.
SFC - solid fat content.

TABLE 6. CONFIRMATORY TEST ON THE SUGGESTED PROCESS CONDITIONS USING ESTABLISHED PROCESS CONDITIONS PLUS HIGH SPEED PIN-ROTOR

		Run: Code:		Cooling perfector	P1	P2	P3
		13	bar	Stage	Auto	Auto	Auto
	High pressure	63.2	°C	P	10.00	10.00	10.00
1	Pump	60	kg hr^{-1}	I	1.00	1.00	1.00
		0.2	kw	D	0.00	0.00	0.00
		20.2	°C				
		0.0	°C	SP	19.0°C	8.0°C	- °C
2	Perfector 1	1 000	rpm/PV	PV	19.1°C	8.4°C	- °C
		0.6	kw	OUT	28%	62%	- %
		35.3	°C	Water heater 80 litres			
		-13.0	°C	SP	55.0°C		
3	Perfector 2	1 000	rpm/PV	PV	54.0°C		
		0.7	kw				
		-	°C	Water heater perfector			
4	Perfector 3	-	°C	SP	45.0°C		
		-	rpm	PV	43.8°C		
			kw	Filling temperature			
5	Pin-rotor	14.8	°C		22.5°C		
	machine	100	rpm				
		0.1	kw	Product: OK			
6	HSPR	1155	rpm				
	DSH pressure i)	3.45	bar				
	ii)	6.89	bar				
	iii)	10.34	bar				

Note: P - proportional. SP - set point.
I - integral. PV - process value.
D - derivative. OUT - out.

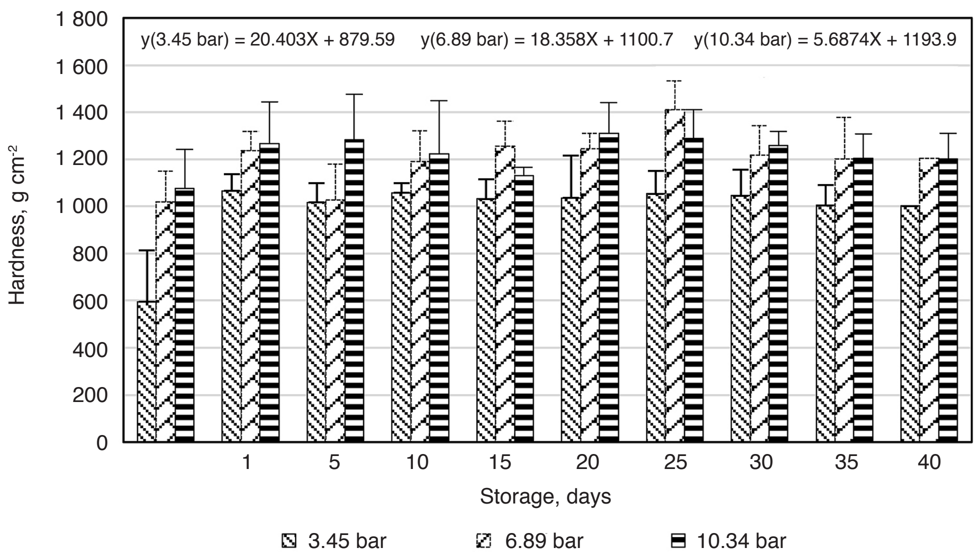


Figure 8. The effects of double stage homogeniser (DSH) pressure of 3.45, 6.89 and 10.34 bar on the hardness of margarine as a function of storage at 5°C.

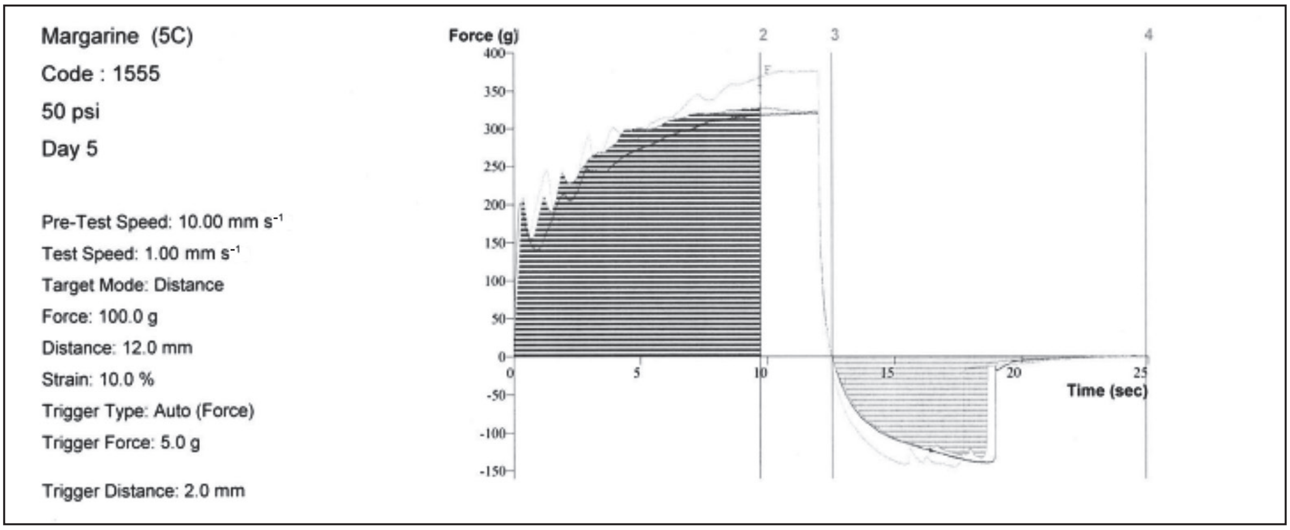


Figure 9. The effects of double stage homogeniser (DSH) pressure of 50 psi (3.45 bar) on the texture of margarine during the fifth day of storage at 5°C. The good texture is shown by the smooth line, while the spreadabilty is determined by the area under curve of 3123 g s^{-1}.

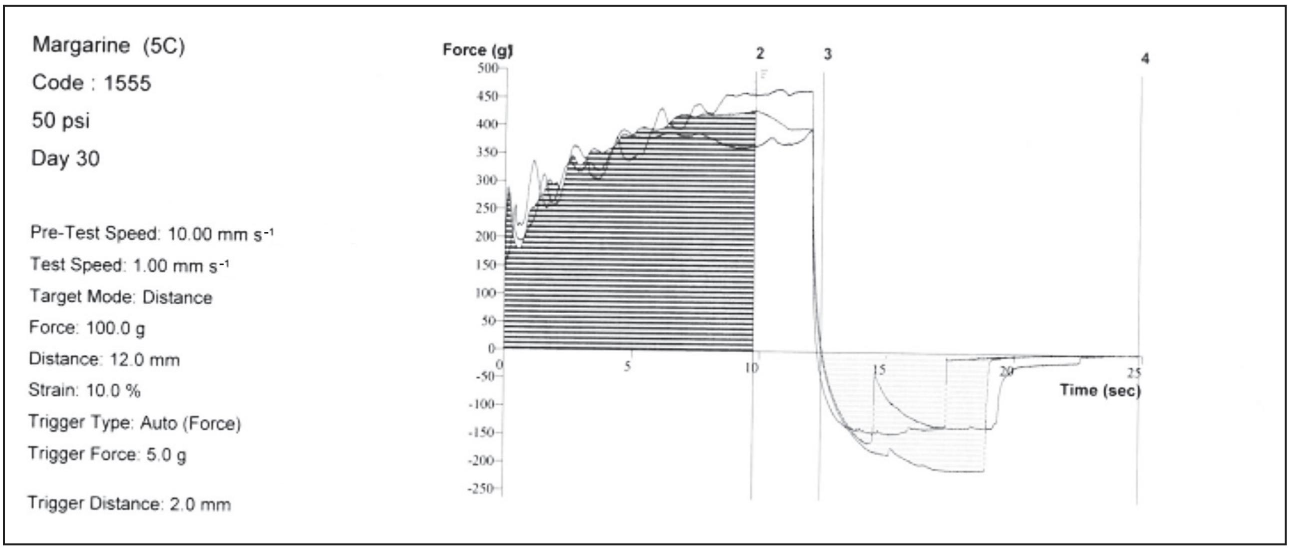


Figure 10. The effects of double stage homogeniser (DSH) pressure of 50 psi (3.45 bar) on the texture of margarine during the 30th day of storage at 5°C. The good texture is shown by the average smooth line, while the spreadabilty is determined by the area under curve of 3668 g s^{-1}.

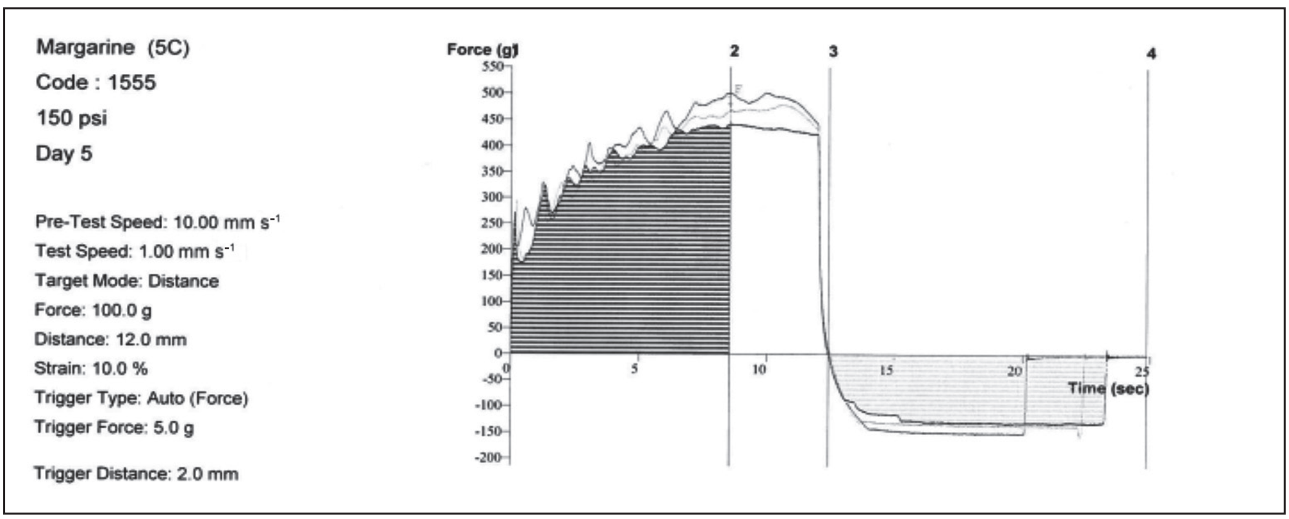


Figure 11. The effects of double stage homogeniser (DSH) pressure of 150 psi (10.34 bar) on the texture of margarine during the fifth day of storage at 5°C. The texture is shown by the sharp line curves, while the spreadabilty is determined by the area under curve of 3471 g s^{-1}.

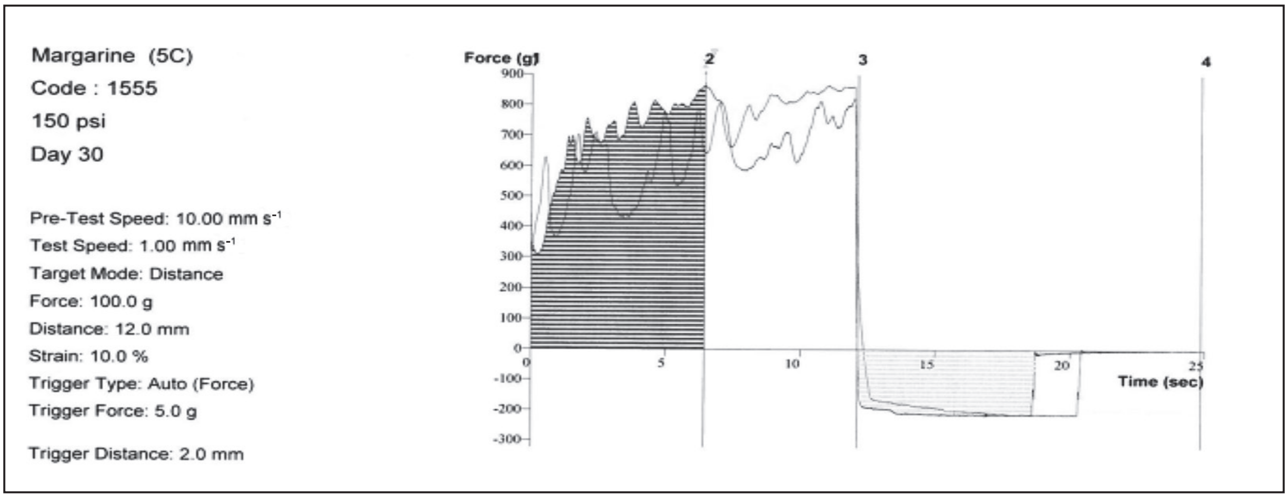


Figure 12. The effects of double stage homogeniser (DSH) pressure of 150 psi (10.34 bar) on the texture of margarine during the 30th day of storage at 5°C. The texture is shown by the sharp line curves, while the spreadabilty is determined by the area under curve of 6743 g s^{-1}.

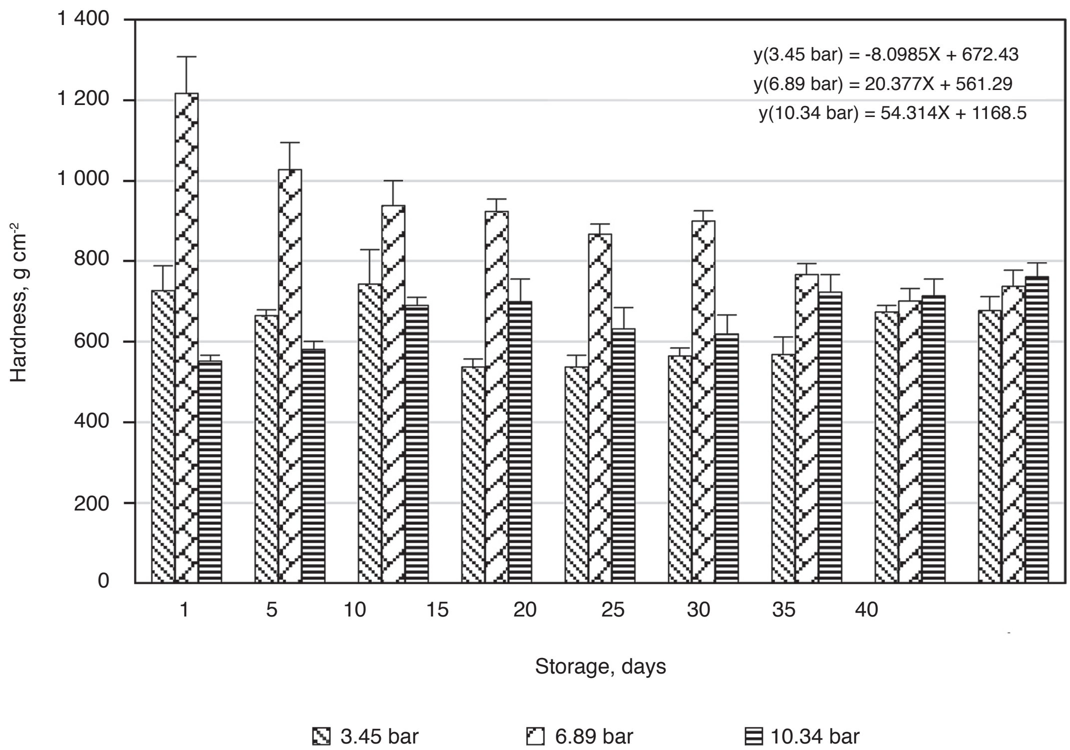


Figure 13. The effects of double stage homogeniser (DSH) pressure of 3.45, 6.89 and 10.34 bar on the hardness of margarine as a function of storage at 10°C.

Storage at 5°C is considered as one of the critical temperatures in soft margarine handling. Producing soft margarine with minimal increase of hardness of <1000 g cm^{-2} with hardening rate of 20.4 g cm^{-2} per week indicates that the suggested HSPR speed and the predicted hardness as well as hardness rate are repeatable. The solid fat development as measured by nuclear magnetic resonance (NMR) at isothermal temperature had shown that the SFC is in the range of 10.1%- 18.1% at 5°C (*Table 7*).

CONCLUSION

Producing soft margarine in either high or low HSPR speed coupled with DSH pressure, could produce the desired margarine texture. However, processing at high HSPR speed will produce a generally hard margarine at 5°C. Medium speed HSPR and low pressure DSH produces the best consistency. The results concluded that HSPR unit coupled with DSH could be added to established margarine

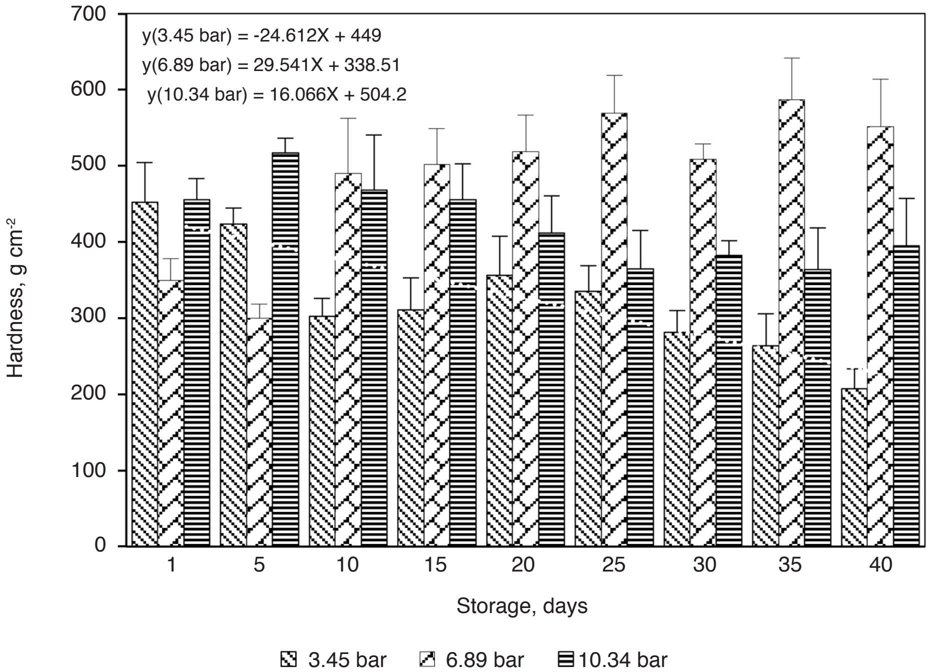


Figure 14. The effects of double stage homogeniser (DSH) pressure of 3.45, 6.89 and 10.34 bar on the hardness of margarine as a function of storage at 15°C.

TABLE 7. SOLID FAT CONTENT DEVELOPED IN THE PRODUCT DURING STORAGE

	Day									
Temp 5°C	0	1	5	10	15	20	25	30	35	40
3.45 bar	14.6	13.4	17.9	15.0	14.2	13.2	13.6	13.8	10.4	10.1
6.89 bar	14.9	17.7	18.4	14.5	14.6	13.6	14.3	14.1	10.1	11.2
10.34 bar	13.7	17.2	17.5	14.3	14.3	13.4	13.8	13.7	10.5	12.2
Temp 10°C										
3.45 bar	-	16.6	16.4	13.1	13.6	13.3	13.3	13.1	11.2	12.6
6.89 bar	-	15.2	16.0	13.4	13.3	13.3	13.5	13.8	10.2	12.1
10.34 bar	-	14.5	15.7	13.0	13.5	13.1	13.1	13.5	10.0	11.9
Temp 15°C										
3.45 bar	-	11.7	11.6	11.8	10.9	10.9	10.9	10.1	8.3	8.3
6.89 bar	-	10.4	11.1	11.3	10.4	10.4	10.0	9.7	8.5	8.4
10.34 bar	-	10.2	11.0	10.9	10.5	10.5	10.4	9.9	7.7	8.3
Temp 20°C										
3.45 bar	-	7.6	7.9	7.9	7.8	7.7	7.6	4.5	4.7	4.0
6.89 bar	-	7.7	7.8	7.9	7.3	7.3	7.2	4.7	4.7	4.2
10.34 bar	-	7.3	7.7	7.6	7.5	7.3	7.1	5.2	5.2	5.2

processing conditions to enhance the completion of crystallisation activities during processing. Post-crystallisation was greatly minimised in the maximum usage of non-hydrogenated palm oil soft margarine formulation at a controlled storage temperature. The findings of this study would also help manufacturers in designing engineering process, processing aspects, conditions, quality control, storage and handling of palm-based products.

RECOMMENDATION

HSPR unit coupled with DSH could be added to an established margarine plant to enhance the completion of crystallisation. Other model studies could be conducted to verify the finding of this study. Further studies that include rheology, polymorphism and microscopy could assist in understanding further the phenomenon of post-crystallisation and the way to handle such a problem.

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