EFFECT OF SOME ADDITIVES ON RESISTANCE TO CRYSTALLIZATION OF PALM OLEIN

Keywords: Additives, crystallization, palm olein, iodine value, cloud point.

NOR AINI IDRIS, YUSOF BASIRON, HANIRAH HASSAN AND ROSZILAH BASSIN*

The effect of certain food additives namely sorbitan tristearate (trade names : Famodan TS and Kemest S65K), polyglycerol esters (THL-3 an THL-9) and lecithin on the resistance to crystallization of palm olein (IV 56, IV 60 and IV 65) during storage at 5°C, 10°C, 15°C and 20°C was studied. The levels of additives added were at 0.01% and 0.10%. Although Famodan TS at 0.10% seemed to promote crystal formation at temperatures of 15°C and below, it in fact delayed crystallization of palm olein at 20°C. Among the additives evaluated, THL-9 showed the best results, as crystal formation was delayed at 5°C, 15°C and 20°C. The addition of THL-9 at 0.10% resulted in a very significant improvement in the stability of palm olein stored at 20°C. Palm olein (IV 56) containing 0.10% THL-9 remained clear for 29 days at 20°C, compared with only 24 hr for the control sample. The addition of lecithin improved the stability of palm olein at 20°C but not at lower temperatures. All the additives studied helped delay crystallization of palm olein at 20°C. With Famodan TS, S65K, THL-3 and THL-9 (at 0.10%) palm olein (IV 65) remained clear for more than 180 days.

INTRODUCTION

Cooking and salad oils are very widely used, both institutionally and domestically. They are prepared from vegetable oils that are usually refined, bleached and deodorized. In some tropi-

*Palm Oil Research Institute of Malaysia

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cal countries, especially Malaysia, palm olein is in common use as a cooking oil.

There is a significant difference between cooking and salad oils. The term 'salad oil' is applied to oils that remain substantially liquid in a refrigerator, that is at about 40°F or 5°C (Swern, 1982). A salad oil should remain bright and clear, whereas a cooking oil may be very cloudy or even solidify under these conditions. Salad oils are used in making mayonnaise and salad dressing.

Clear, light-coloured oils of vegetable origin with good oxidative and cold stability are preferred for salad oils, and while palm olein has an attractive appearance and good oxidative stability, it becomes cloudy and tends to crystallize in cold weather in temperate countries. This problem might be partly solved by using appropriate additives, to delay or prevent crystallization at low temperatures. Thus the objective of the present study was to examine the effect of some additives on the resistance to crystallization of palm olein.

MATERIALS AND METHODS

Refined, bleached and deodorized palm olein of Iodine Value (IV) 56 (single fractionated), IV 60 (double fractionated) and IV 65 (double fractionated) were obtained from Lam Soon (M) Bhd., Petaling Jaya. The additive Famodan TS was obtained from Grinsted, Denmark; Kemest S65K was supplied by Harrisons Trading (Peninsular) Sdn. Bhd. Petaling Jaya; THL-3 and THL-9 were supplied by Sakamoto Yakuhin Kogyo Co., Ltd. Osaka, Japan; and lecithin was obtained from Behn Mayer, Kuala Lumpur.

Famodan TS is a trade name for sorbitan tristearate. It is in the form of tiny white beads. Kemest S65K is also a sorbitan tristearate but it has a creamy colour and comes in the form of slightly larger beads than Famodan TS. THL-3 is a type of polyglycerol ester, in the form of colourless fluid while THL-9, a type of polyglycerol ester, comes in the form of white solid.

About 10 kg oil was filtered using 24 cm Whatman qualitative filter papers (Whatman International Ltd., England) and filter funnels (top internal diameter : 9.5 cm, stem length : 10 cm) in a warming cabinet at 60°C. Then, 10 beakers (1500 ml) labelled with the names of the additives (Famodan TS, S65K, THL-3, THL-9 and lecithin) at two different levels (0.01% and 0.10% by weight of the oil) were prepared. Filtered oil (1000 ml) was poured into each beaker and heated to 130°C. Additives were put into the oil in each beaker and mixed in thoroughly. Samples of 140 g of each oil were poured into six plastic bottles at 60°C. The samples were allowed to cool at room temperature before four of them were stored at different temperatures 5°C, 10°C, 15°C and 20°C. The samples in the other two bottles were used for cold test determinations and other analyses. Observations were conducted daily to determine how long the samples remained clear.

The cold test at 0°C was carried out according to AOCS method, No. Cc 11-53 (AOCS, 1987). Cloud point was determined according to AOCS Method No. Cc 6-25 (AOCS, 1987). The tempering temperatures used in determining the cloud points were as in *Table 1*.

RESULTS AND DISCUSSION

Table 2 shows that palm olein of IV 56 remained clear longer (24 hr) when stored at 20°C than samples stored at 5°C (2 hr), 10°C or 15°C (> 3 hr). The addition of Famodan TS at 0.01% and 0.10% delayed crystal formation during storage at 20°C to 3 days and 4 days, respectively. However, there was no improvement in resistance to crystallization of the palm olein at lower temperatures of 5°C, 10°C or 15°C with the addition of Famodan TS (*Figure 1*). At lower temperatures 5° and 10°C, S65K seemed to promote crystallization of single fractionated palm olein (IV 56). However, at 0.10%, it delayed crystal formation in a sample kept at 15°C to more than 24 hr as compared with crystallization in slightly more than 3 hr in the control sample. Like Famodan TS, S65K also improved stability of palm olein (IV 56) at 20°C, with the 0.10% level showing a better effect than 0.01 per cent.

Although the additive THL-3 at 0.01% improved the resistance to crystallization of palm olein (IV 56) at 15°C (< 24 hr) and at 20°C (2 days), it actually made the olein less stable at a storage temperature of 5°C, with the sample remaining clear for only 1.5 hours. Nonetheless, addition of THL-3 at 0.10% gave better results, particularly at

TABLE 1. TEMPERATURES (°C) AT WHICH SAMPLES WERE TEMPERED FOR CLOUD POINT DETERMINATION

		Palm Olein		
Additives	**Level added (%)**	**IV 56**	**IV 60**	**IV 65**
Control		1.0	-1.5	-3.5
Famodan TS	0.01	1.0	0.0	-2.0
	0.10	1.5	0.0	-2.5
S65K	0.01	1.5	-1.5	-3.5
	0.10	2.0	-1.5	-2.0
THL-3	0.01	1.0	0.0	-3.5
	0.10	1.5	0.0	-3.5
THL-9	0.01	1.0	0.0	-2.0
	0.10	2.0	1.0	-2.5
Lecithin	0.01	1.5	-1.5	-3.5
	0.10	1.5	-1.5	-3.5

15°C (24 hr) and 20°C (4 days). Better results were obtained with the addition of lecithin compared with the control sample or with Famodan TS, S65K or THL-3. In the case of lecithin, the results were the same for 0.01% and 0.10 per cent. Among all the additives evaluated, THL-9 showed the best results, as crystal formation was delayed at 5°C, 15°C and 20°C. The addition of THL-9 at 0.10% resulted in a very significant improvement in the stability of palm olein (IV 56) stored at 20°C (*Figures 2* and *3*). The results indicated that, in general, the additives produced better effects at a concentration of 0.10% than at 0.01 per cent. A sample containing 0.10% THL-9 remained clear for 29 days at 20°C, compared with only 24 hr for the control sample (*Table 2*).

The resistance to crystallization of double fractionated palm olein (IV 60) was better than that of single fractionated palm olein (IV 56) at all temperatures studied (*Table 3, cf. Table 2*). At 15°C and 20°C, the olein remained clear for one whole day and 7 days, respectively, compared with only slightly more than 3 hr (at 15°C) and 24 hr (at 20°C) for single fractionated palm olein.

The addition of Famodan TS at 0.01% and 0.10% actually resulted in faster crystallization at 5°C, 10°C and 15°C than in the control sample at similar storage temperatures. *Figure 4* shows the appearance of the samples (palm olein, IV 60) stored at 15°C at day 30. The addition of Famodan TS, however, was beneficial in the sample stored at 20°C. Very good stability was observed in the sample containing 0.10% Famodan TS: 49 days against 10 days in the sample containing 0.01 per cent.

Faster crystallization occurred in samples containing S65K at 5°C and 10°C, than in the control samples. However, S65K, at 0.10% promoted crystallization at a slower rate than did 0.10% Famodan TS. Addition of S65K at 0.10% was beneficial in samples stored at 15°C and 20°C (*Figures 4* and *5*), as crystallization was delayed for 7 days and 49 days, respectively (*Table 3*).

Addition of THL-3 at 0.01% delayed crystal formation at 20°C (to 10 days) but not at lower temperatures. Crystal formation was slightly delayed at 5°C and 15°C to > 5 hr and 3 days, respectively, by the addition of THL-3 at 0.10 per

cent. However, it did improve stability of palm olein (IV 60) stored at 20°C to 39 days (compared with 7 days in the control sample). It was noted that THL-9 at 0.01% and 0.10% promoted crystallization of palm olein (IV 60) stored at 5°C : crystals were formed within 1.5 hours. However, at 0.01%, THL-9 delayed crystal formation at storage temperatures of 15°C and 20°C to 3 days and 53 days, respectively (*Table 3*). The addition of THL-9 at 0.10% produced better results than at 0.01%, at storage temperatures of 15°C and 20°C (*Figure 6*). At 15°C and 20°C, palm olein (IV 60) containing 0.10% THL- 9 remained clear for 24 and 60 days respectively (*Table 3*). The addition of lecithin improved the stability of palm olein at 20°C but not at lower temperatures.

The stability of double fractionated palm olein, IV 65, (*Table 4*) was better than that of double fractionated palm olein, IV 60, (*Table 3*) or single fractionated palm olein, IV 56 (*Table 2*). At 15°C, it remained clear for 3 days compared with only 1 day and more than 3 hr, in the case of palm oleins, of IV 60 and IV 56, respectively. At 20°C the double fractionated palm olein of IV 65 remained clear for 59 days, compared with only 7 days in the case of palm olein of IV 60, and 3 days in the case of single fractionated palm olein (IV 56). Double fractionated palm olein of IV 65 formed crystals at a slower rate than did double fractionated palm olein of IV 60 or single fractionated olein of IV 56 (*Tables 2* to *4*).

Famodan TS at 0.01% improved the resistance to crystallization of double fractionated palm olein of IV 65 at temperatures of 10°C and higher. However, the higher level of Famodan TS, 0.10%, seemed to promote crystal formation at temperatures of 15°C and lower. Therefore, a lower level of Famodan (0.01%) would be recommended for better cold stability of palm olein of any IV at temperatures of 15°C and below. The additives did not have much effect on the crystallization of double fractionated palm olein of IV 65 at the storage temperature of 5°C. However, it was noted that Famodan TS at 0.10% promoted crystal formation at both 5°C and 10°C within 0.5 hour.

At 10°C, certain additives did help delay crystal formation (*Figure 7* and *Table 4*). These included Famodan TS at 0.01% (1 day), THL-3 at both 0.01% (1 day) and 0.10% (2 days), THL-9 at both levels (1 day each) and lecithin at 0.10% (2 days). On the other hand, S65K at both 0.01% and 0.10% failed to delay crystallization during storage at 10°C. Samples containing S65K formed crystals within 4.5 hours. (Crystals were formed in the control sample at 10°C in less than one day.) Much better stability was observed at higher temperatures 15°C and 20°C (*Figures 8* and *9*). Among the additives studied, THL-9 was the most effective in delaying crystallization at 15°C. With the addition of THL-9 at 0.01% and 0.10%, the oil remained clear at 15°C for 23 and 60 days, respectively (*Table 4*), compared with only 3 days for the control sample. The second most effective additive was THL-3, which at the levels of 0.01% and 0.10%, kept the oil clear for 11 and 18 days respectively.

Figures 10 and *11* show the appearance of palm olein of IV 65 containing 0.01% and 0.10% additives, kept at 20°C, at day 40 and day 45, respectively. All samples, including the control, remained clear at 20°C at days 40 and 45. In fact, with most of the additives (at 0.10%), palm olein (IV 65) remained clear for more than 180 days (*Table 4, Figure 12*): thus all the additives studied helped delay crystallization of palm olein (IV 65) at 20°C. At 15°C, all the additives (with the exception of Famodan TS at 0.10%) also helped delay crystallization of palm olein (IV 65).

The standard method of evaluating salad oil is called the 'cold test', which is used to determine the ability of the oil to withstand refrigeration. An oil is considered to pass the minimum test if it remains perfectly clear after standing for 5.5 hours at 0°C (Weiss, 1980). All the oil samples in the present work failed the cold test at 0°C as they became cloudy in less than 5.5 hours.

In general, the use of additives with single fractionated palm olein of IV 56 resulted in a slight increase in the cloud point of the samples (*Table 5*). There were also slight increases in the cloud points of palm olein (IV 60) containing Famodan TS (at 0.01 and 0.10%), THL-3 (at 0.10%) and THL-9 (at 0.01%). On the other hand, the addition of lecithin at 0.01% and 0.10% and of S65K at 0.01% resulted in slight reductions in the cloud points of the samples. Although there were increases in the cloud point of double fractioned palm olein of IV 65 with the addition of Famodan TS (at both 0.01% and 0.10%), S65K (at 0.10%), and THL-9 (at 0.01%), the addition of THL-3 or leci-

TABLE 2. RESISTANCE TO CRYSTALLIZATION[a] OF SINGLE FRACTIONATED PALM OLEIN OF IV 56 WITH ADDITIVES AT 0.01% AND 0.10%, AT STORAGE TEMPERATURES OF 5°C, 10°C, 15°C AND 20°C

Additives	Level added (%)	Temperature (°C)			
		5	10	15	20
Control		2 hr	> 3 hr	> 3 hr	24 hr
Famodan TS	0.01	2 hr	2 hr	> 3 hr	3 days
	0.10	0.5 hr	1 hr	1.5 hr	4 days
S65K	0.01	1.5 hr	1.5hr	> 3 hr	3 days
	0.10	1.5 hr	1.5 hr	> 24 hr	8 days
THL-3	0.01	1.5 hr	> 3 hr	< 24 hr	2 days
	0.10	2.5 hr	> 3 hr	24 hr	4 days
THL-9	0.01	> 3 hr	> 3 hr	< 24 hr	4 days
	0.10	> 3 hr	> 3 hr	4 days	29 days
Lecithin	0.01	> 3 hr	> 3 hr	24 hr	4 days
	0.10	> 3 hr	> 3 hr	24 hr	4 days

[a] Time the oil remained clear.

TABLE 3. RESISTANCE TO CRYSTALLIZATION[a] OF DOUBLE FRACTIONATED PALM OLEIN OF IV 60 WITH ADDITIVES AT 0.01% AND 0.10% AT STORAGE TEMPERATURES OF 5°C, 10°C, 15°C AND 20°C

Additives	Level added (%)	Temperature (°C)			
		5	10	15	20
Control	0.10	3 hr	> 5 hr	1 day	7 days
Famodan TS	0.01	1.5 hr	3 hr	< 24 day	10 days
	0.10	0.5 hr	0.5 hr	1 hr	49 days
S65K	0.01	1.5 hr	2.0 hr	5 hr	10 days
	0.10	1.5 hr	1.0 hr	7 days	49 days
THL-3	0.01	2.5 hr	> 5 hrs	1 day	10 days
	0.10	> 5 hr	> 5 hrs	3 days	39 days
THL-9	0.01	1.5 hr	> 5 hrs	3 days	53 days
	0.10	1.5 hr	> 5 hrs	24 days	60 days
Lecithin	0.01	3 hr	> 5 hr	1 day	28 days
	0.10	1.5 hr	> 5 hr	1 day	28 days

[a] Time the oil remained clear.

TABLE 4. RESISTANCE TO CRYSTALLIZATION[a] OF DOUBLE FRACTIONED PALM OLEIN OF IV 65 WITH ADDITIVES AT 0.01% AND 0.10% AT STORAGE TEMPERATURES OF 5°C, 10°C, 15°C AND 20°C

Additives	Level added (%)	Temperature (°C)			
		5	10	15	20
Control		> 4.5 hr	> 5 hr	3 days	59 days
Famodan TS	0.01	> 4.5 hr	1 day	7 days	> 150 days
	0.10	0.5 hr	0.5 hr	2 hr	> 180 days
S65K	0.01	4.5 hr	4.5 hr	8 days	< 115 days
	0.10	4.5 hr	4.5 hr	11 days	> 180 days
THL-3	0.01	> 4.5 hr	1 day	11 days	< 150 days
	0.10	> 4.5 hr	2 days	18 days	> 180 days
THL-9	0.01	> 4.5 hr	1 day	23 days	> 180 days
	0.10	> 4.5 hr	1 day	60 days	> 180 days
Lecithin	0.01	> 4.5 hr	> 4.5 hr	7 days	> 150 days
	0.10	> 4.5 hr	2 days	11 days	> 150 days

[a] Time the oil remained clear.

TABLE 5. CLOUD POINTS (°C) OF PALM OLEIN OF IV 56, IV 60, IV 65 AND SAMPLES CONTAINING VARIOUS ADDITIVES AT 0.01 % AND 0.10 PER CENT.

Additives	Level added (%)	Cloud Points of Palm Olein		
		IV 56	IV 60	IV 65
Control		6.0	4.7	2.5
Famodan TS	0.01	6.0	5.0	3.3
	0.10	6.2	5.0	3.5
S65K	0.01	6.3	4.5	2.5
	0.10	7.0	4.8	3.0
THL-3	0.01	6.3	4.8	2.5
	0.10	6.4	5.0	2.5
THL-9	0.01	6.4	5.3	2.9
	0.10	6.5	4.5	2.5
Lecithin	0.01	6.5	4.5	2.5
	0.10	6.5	4.5	2.5

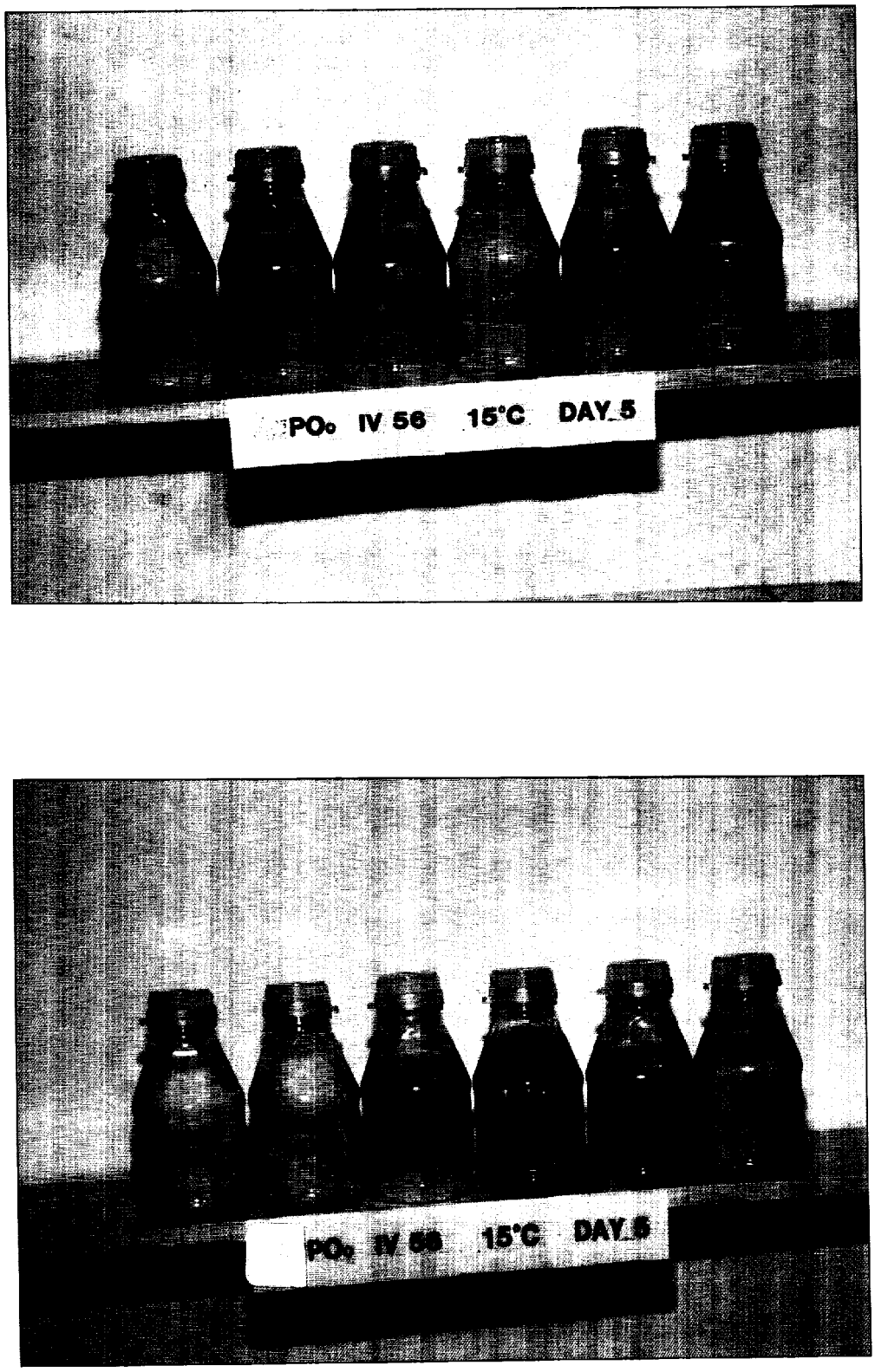
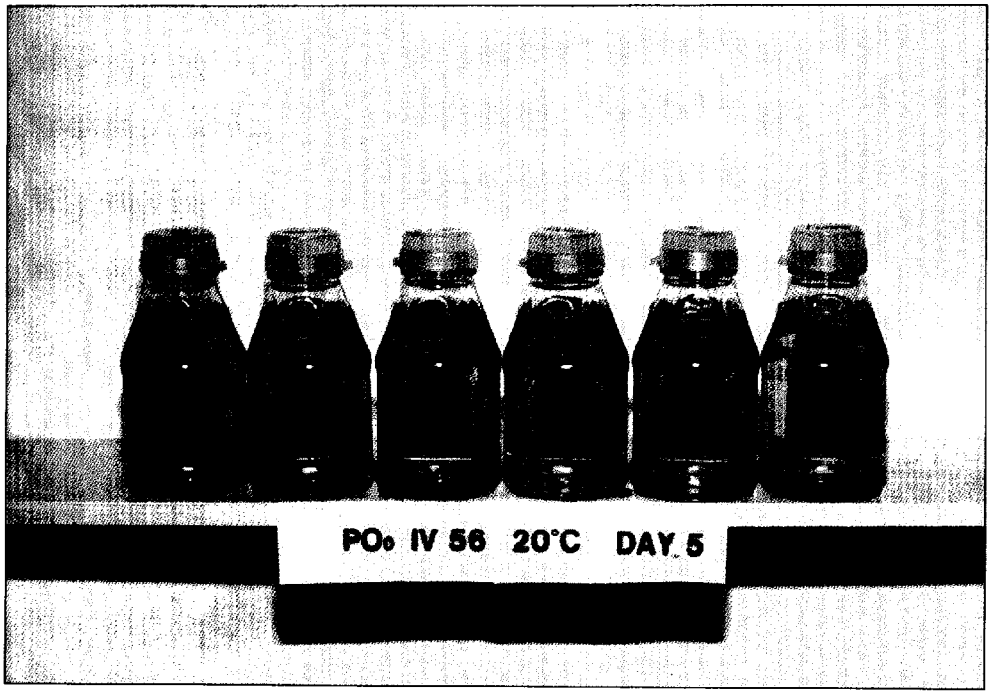


Figure 1. Appearance of single fractionated palm olein of iodine value 56 with additives at a) 0.01% and b) 0.10%, stored at 15°C for 5 days.



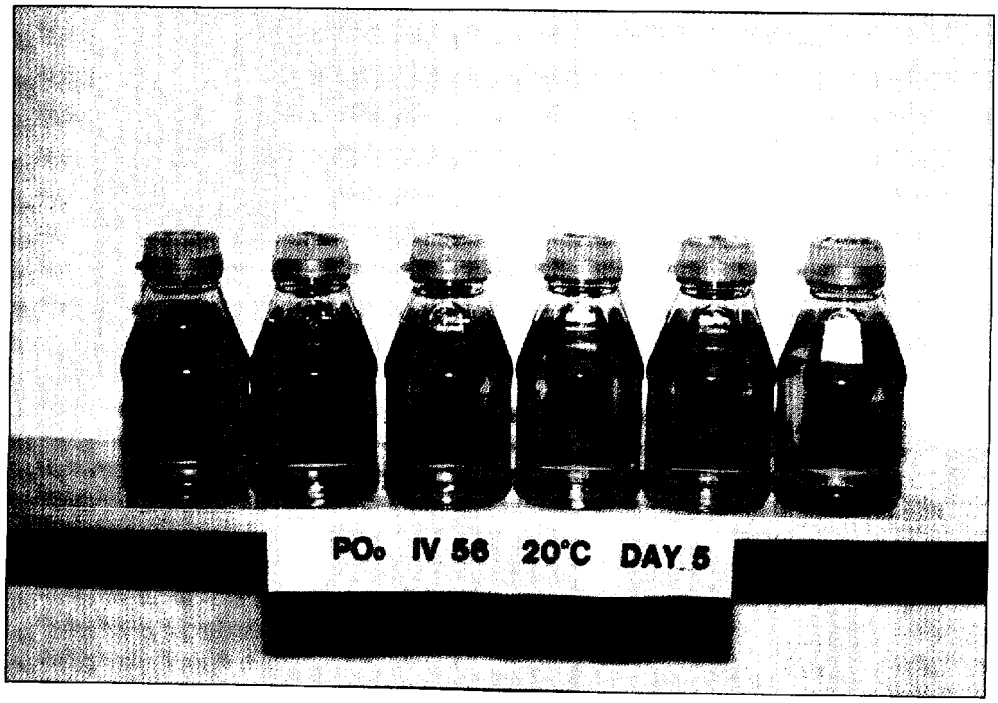
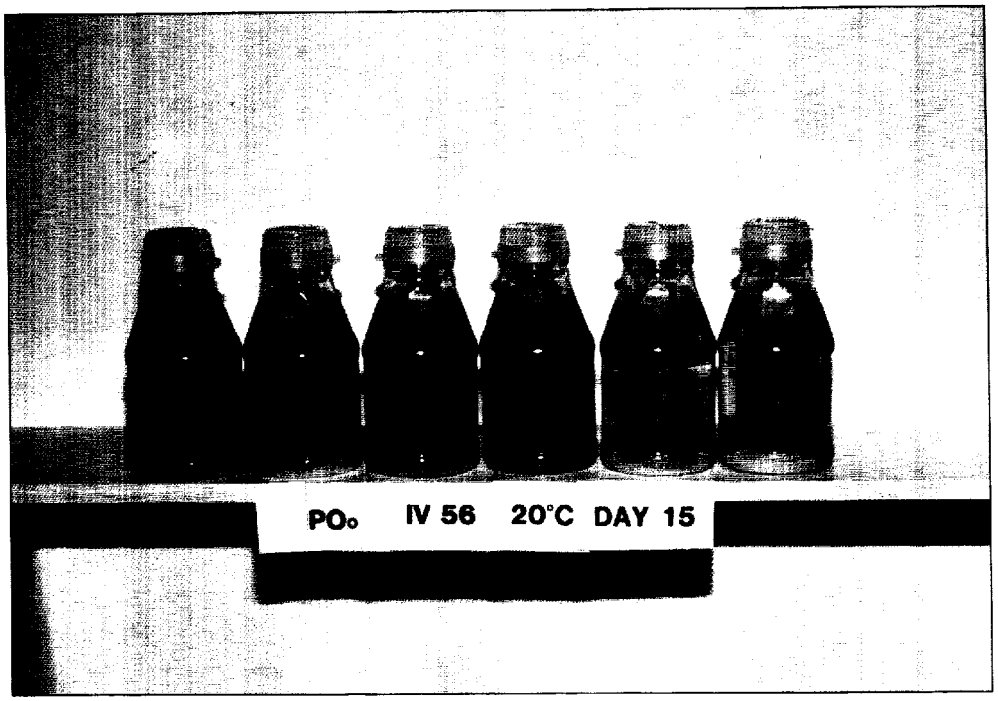


Figure 2. Appearance of single fractionated palm olein of iodine value 56 with additives at a) 0.01% and b) 0.10%, stored at 20°C for 5 days.



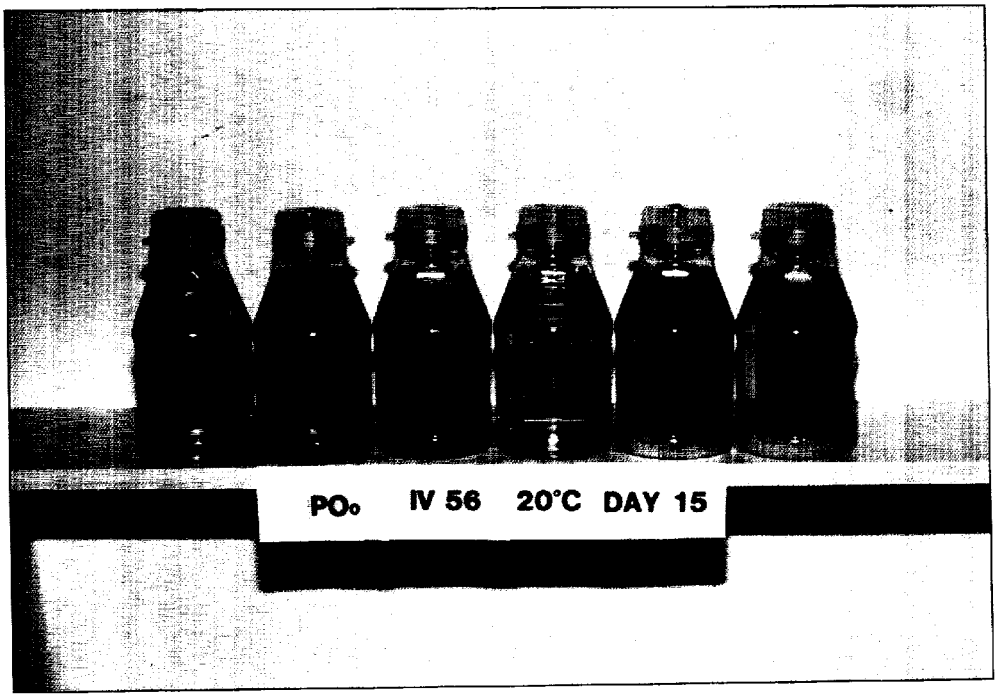
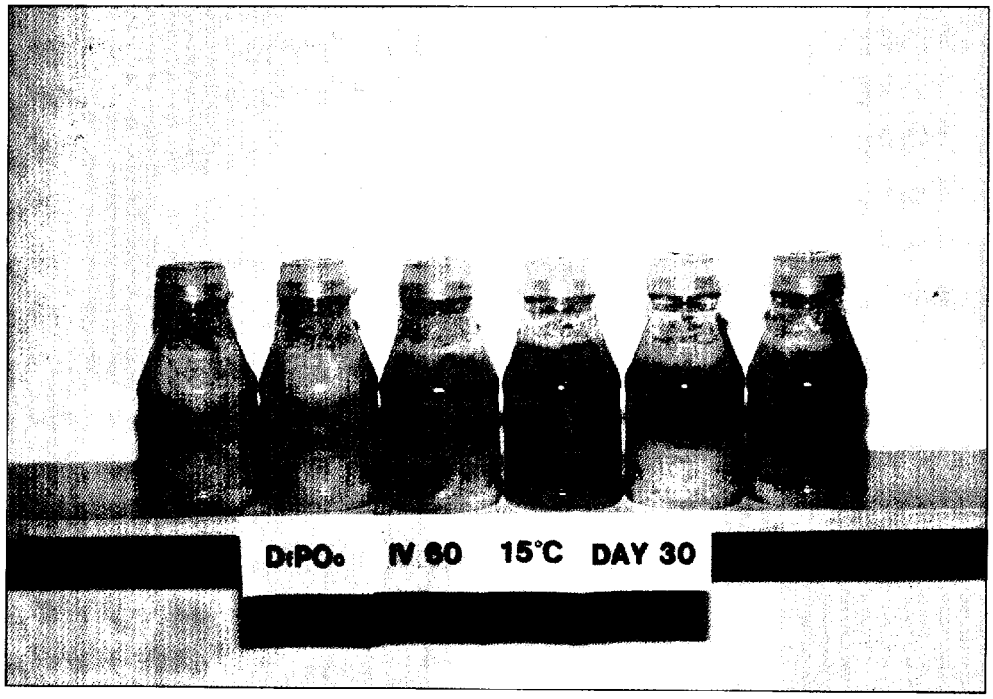


Figure 3. Appearance of single fractionated palm olein of iodine value 56 with additives at a) 0.01% and b) 0.10%, stored at 20°C for 15 days.



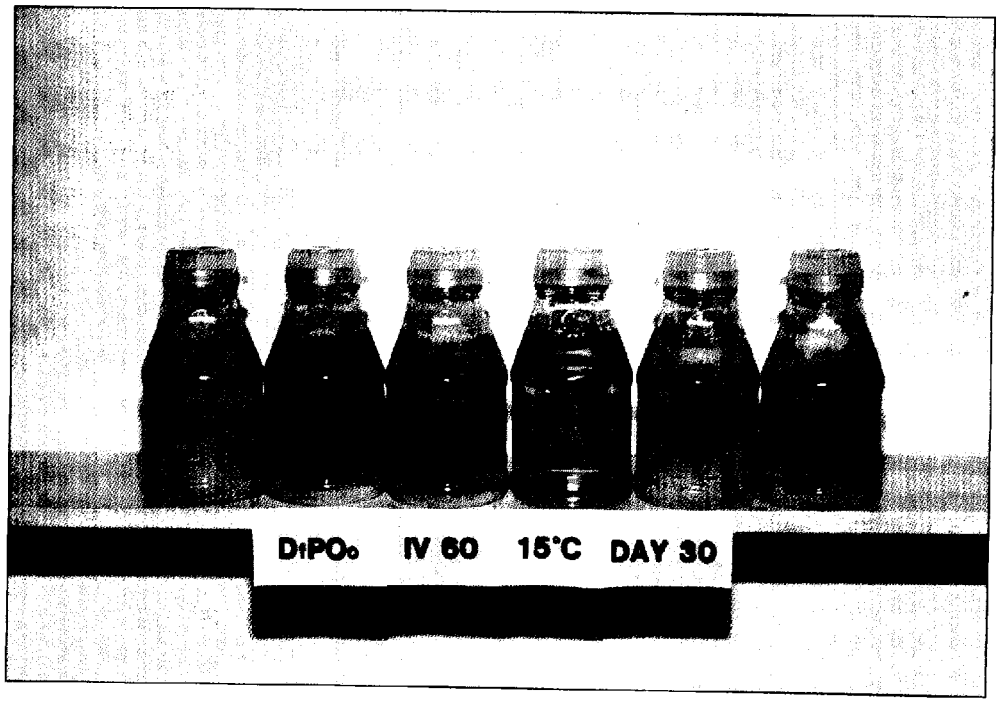


Figure 4. Appearance of double fractionated palm olein of iodine value 60 with additives at a) 0.01% and b) 0.10%, stored at 15°C for 30 days.

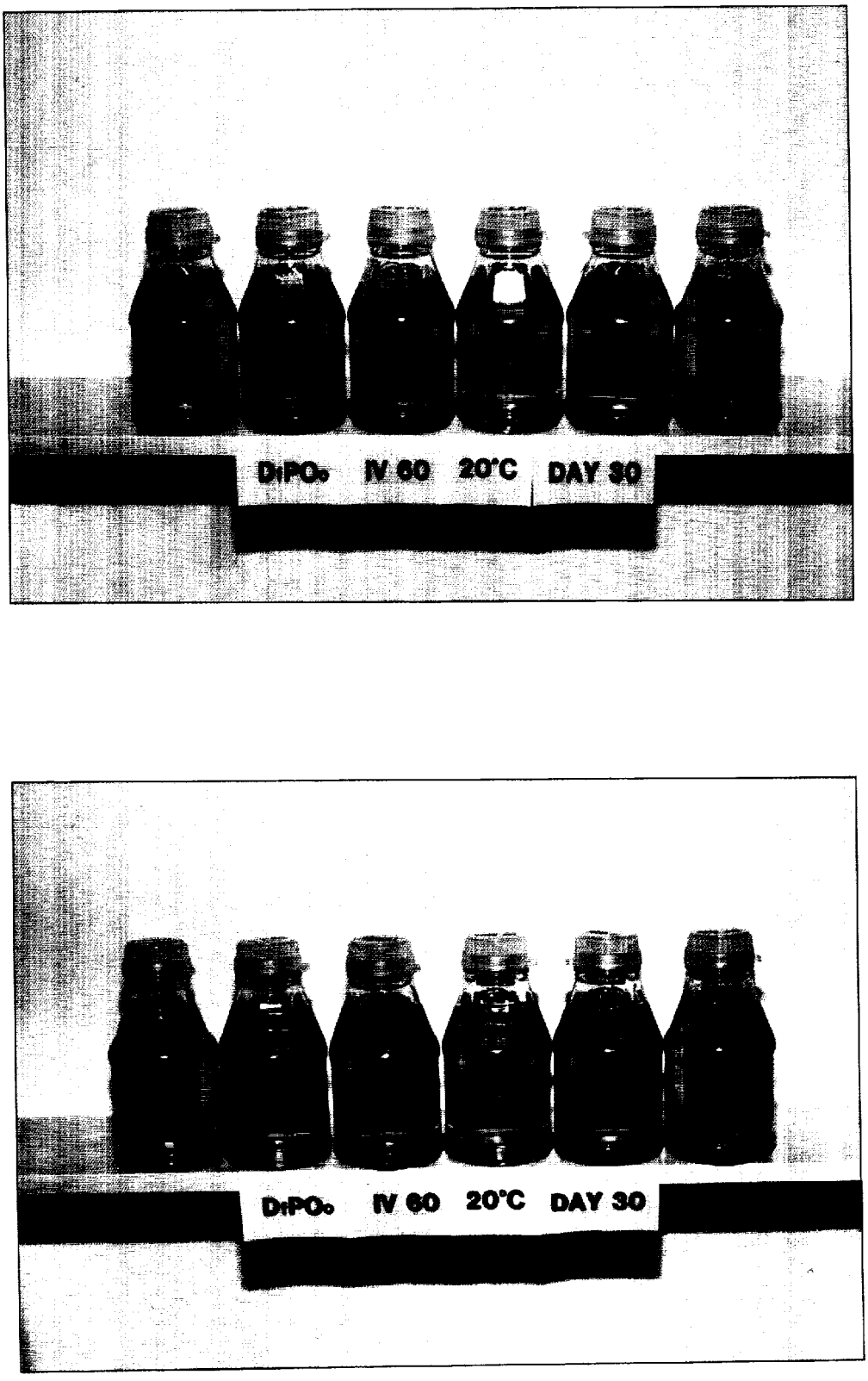
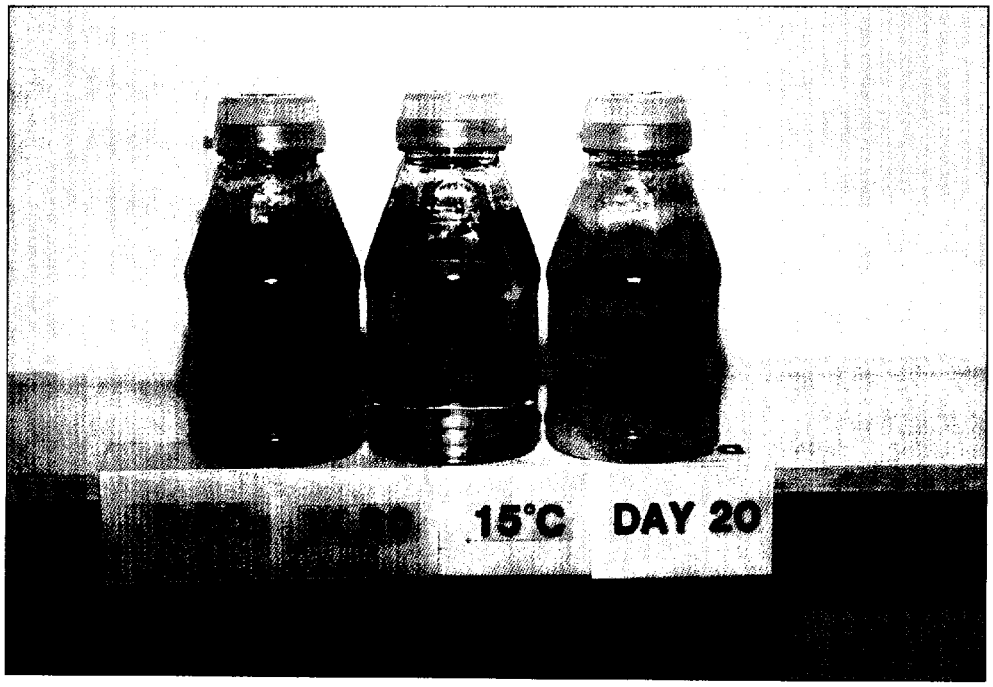


Figure 5. Appearance of double fractionated palm olein of iodine value 60 with additives at a) 0.01% and b) 0.10%, stored at 20°C for 30 days.



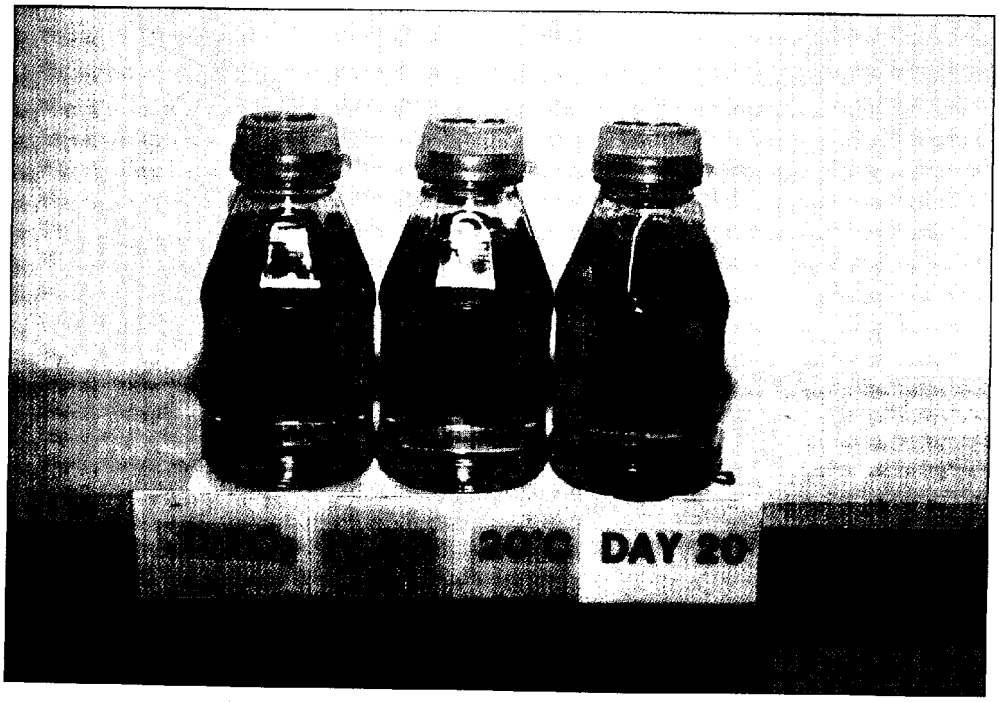
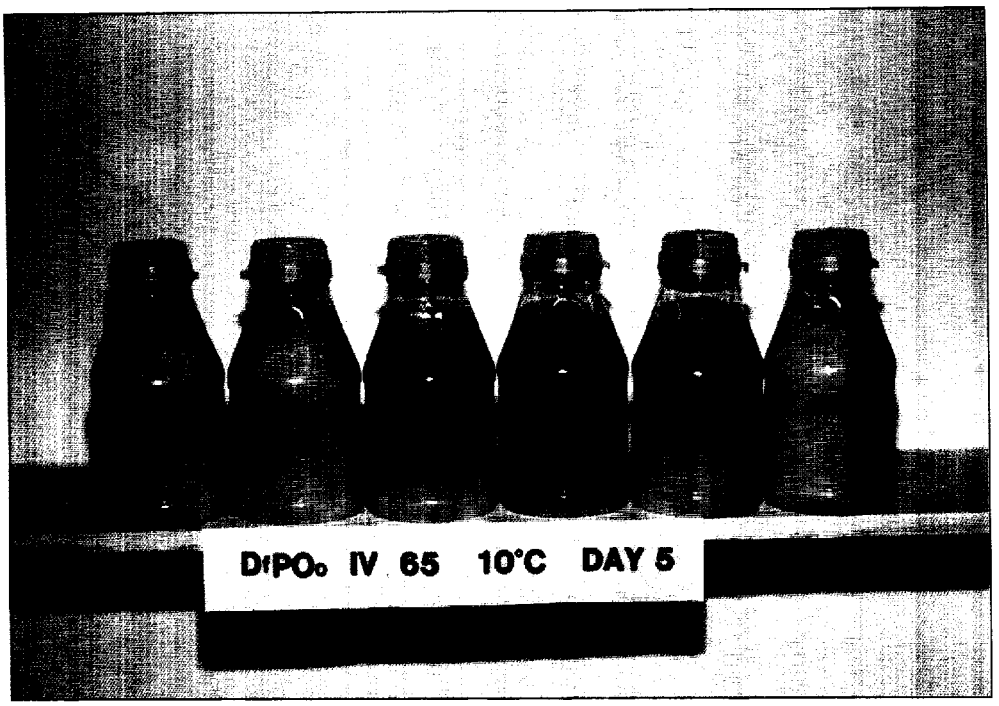


Figure 6. Comparison between cold stability of double fractionated palm olein of iodine value 60 containing THL-9 at 0.01% and 0.10% at day 20, stored at a) 15°C and b) 20°C.



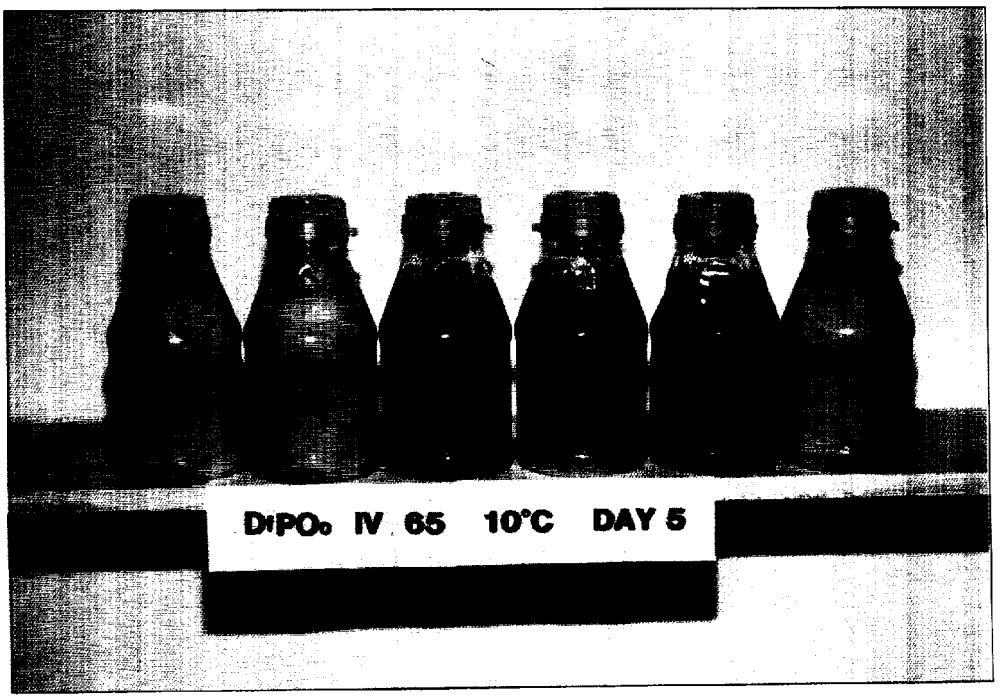
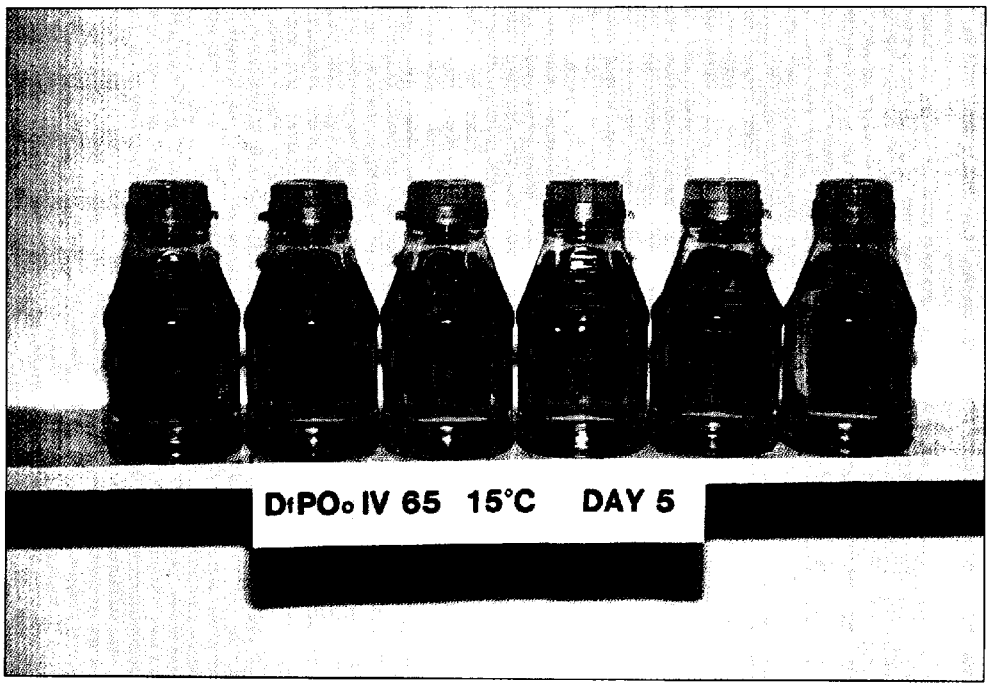


Figure 7. Appearance of double fractionated palm olein of iodine value 65 containing additives at a) 0.01% and b) 0.10%, stored at 10°C for 5 days.



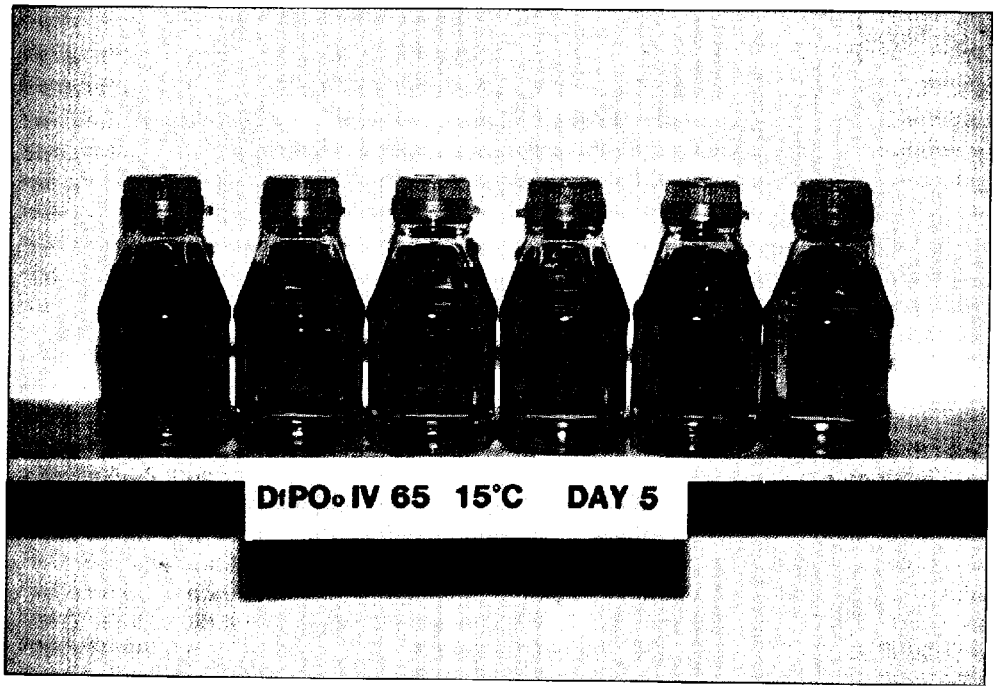
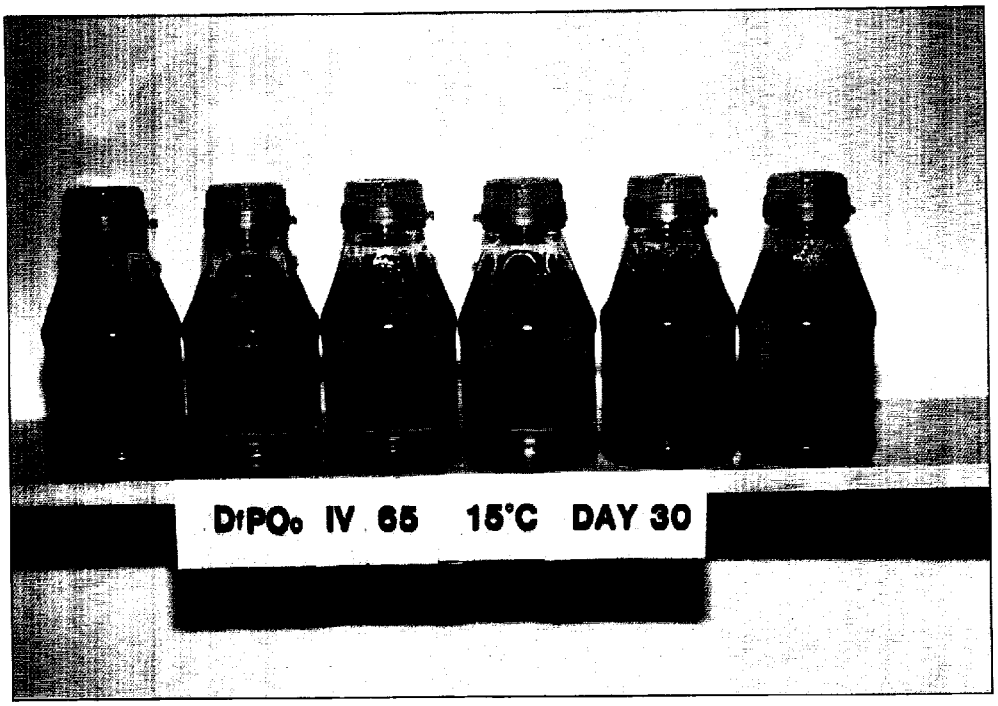


Figure 8. Appearance of double fractionated palm olein of iodine value 65 containing additives at a) 0.01% and b) 0.10%, stored at 15°C for 5 days.



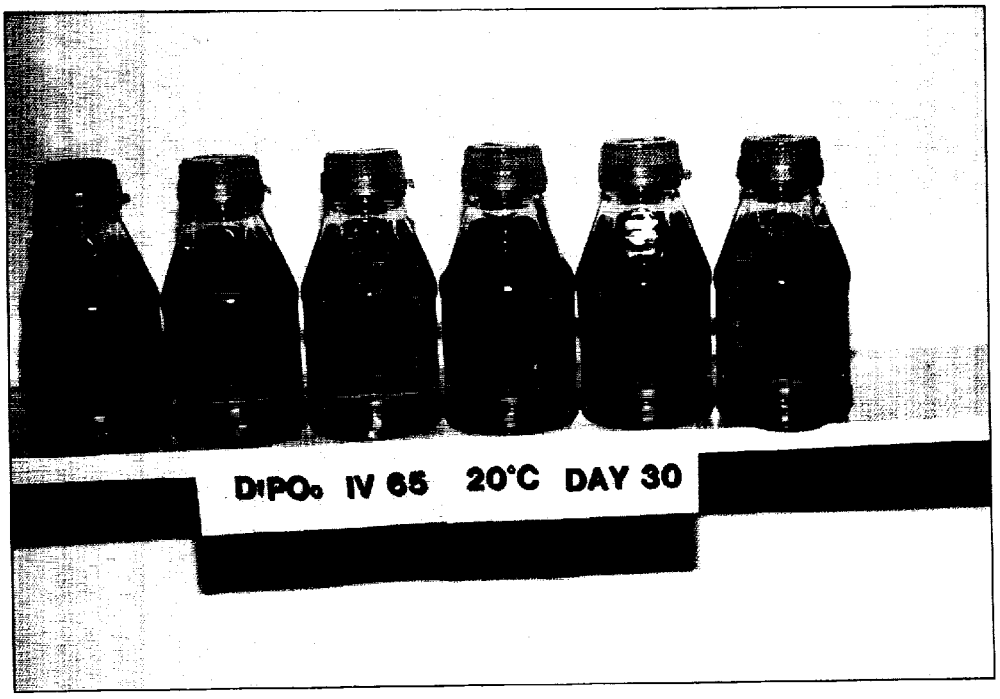
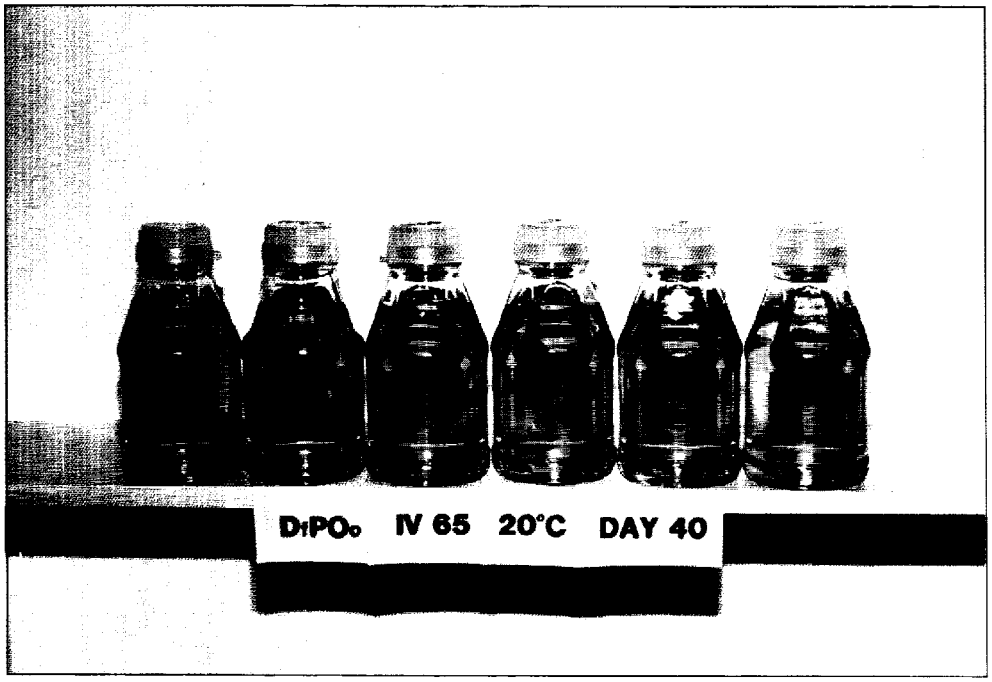


Figure 9. Appearance of double fractionated palm olein of iodine value 65, containing additives at 0.10%, stored at a) 15°C and b) 20°C for 30 days.



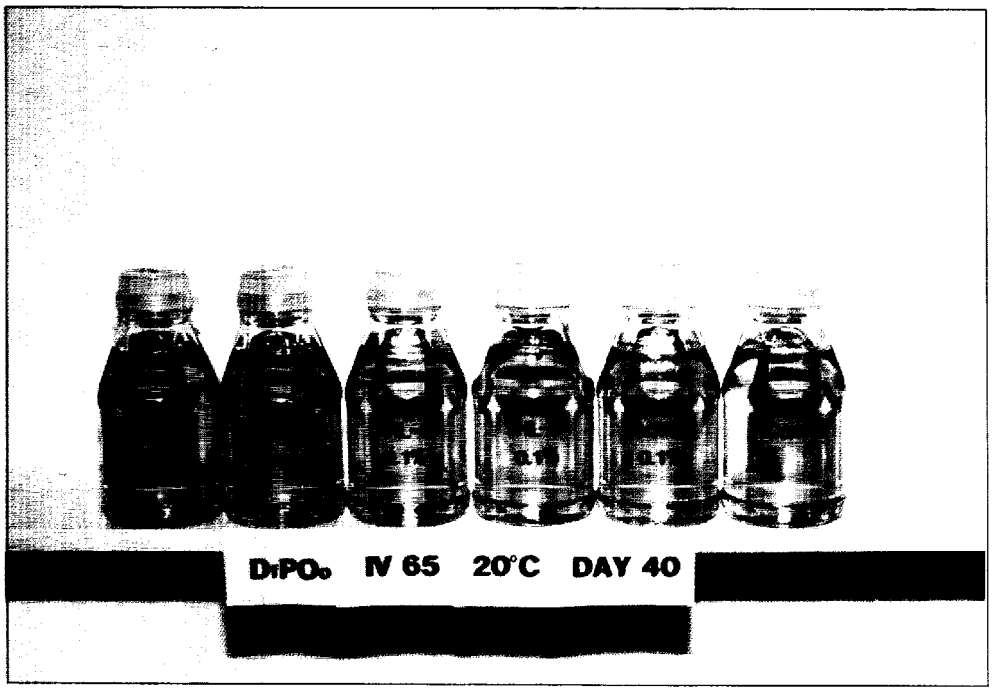
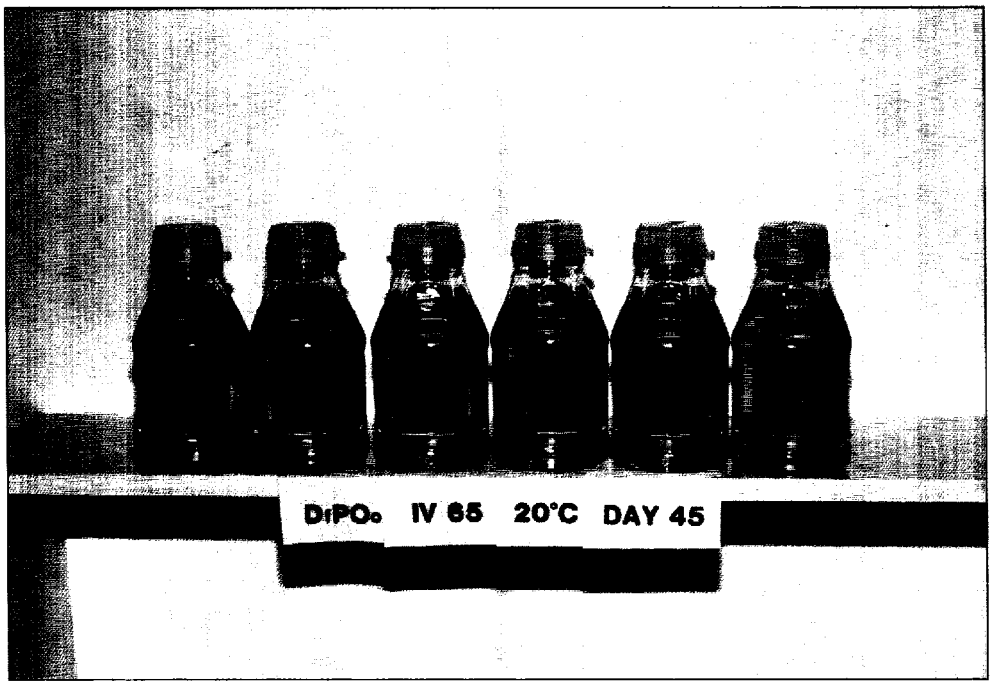


Figure 10. Appearance of double fractionated palm olein of iodine value 65 with additives at a) 0.01% and b) 0.10%, stored at 20°C for 40 days.



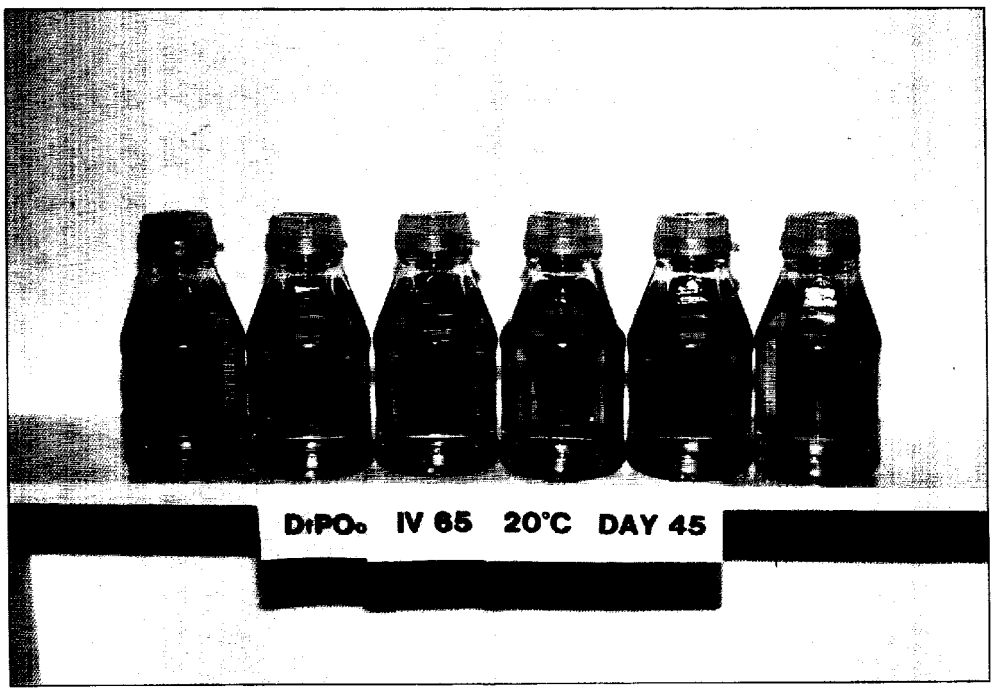


Figure 11. Appearance of double fractionated palm olein of iodine value 65 with additives at a) 0.01% and b) 0.10%, stored at 20°C for 45 days.

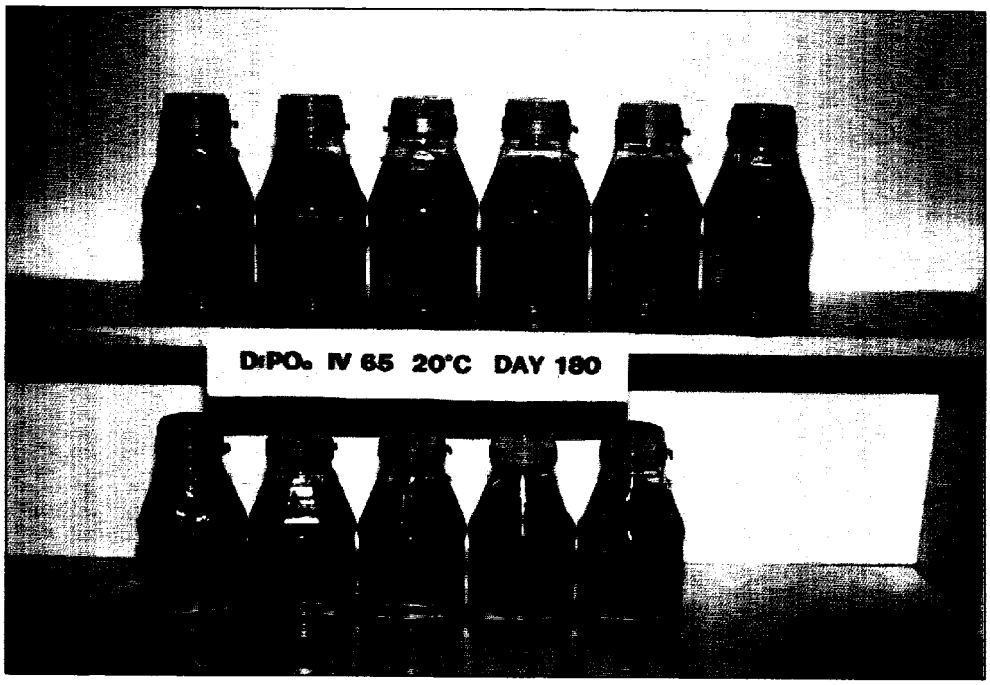


Figure 12. Appearance of double fractionated palm olein of iodine value 65 with additives at 0.01% and 0.10%, stored at 20°C for 180 days.

thin had no effect. The results of the study suggested that there was no direct relationship between the cloud points of the samples which contained additives and their resistance to crystallization at various storage temperatures.

CONCLUSIONS

Although Famodan TS delayed crystallization of palm olein at 20°C, at the higher level of 0.10%, it seemed to promote crystal formation at temperatures of 15°C and below. Crystallization of single fractionated palm olein (IV 56) and double fractionated palm olein (IV 65) at both 15°C and 20°C was delayed by the addition of S56K, THL-3, THL-9 and lecithin at either the 0.01% or the 0.10% level. However, at the lower temperature of 5°C, certain additives seemed to promote crystallization of palm olein. THL-9 was found to be the most effective among all the additives studied, in delaying crystallization of palm olein at a storage temperature of 20°C. It was found to be more effective at the higher level of 0.10% than at 0.01 per cent. Thus the study implied that additives could be applied to cooking oil where the effect is beneficial in delaying crystal formation at ambient temperature in temperate countries or at air-conditioned market shelf temperature in tropical countries. However, they may not be suitable for application in salad oil or in oil to be used in making mayonnaise, as the product needs to be refrigerated.

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