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Changes of Fatty Acid Composition of Lipid in Raw and Processed Adlay Powder during Storage

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Abstract

Raw adlay powder (RAP) and processed adlay powder (PAP) were prepared and the changes of fatty acid compositions of lipids in RAP and PAP during storage at 5 °C and 35 °C for six months were studied. The major fatty acids found in the adlay lipids were oleic acid (28-45%), linoleic acid (38-50%) and palmitic acid (14-18%). Throughout the storage period, the concentrations of linoleic acid were decreased in samples stored at 35 °C, but those of oleic acid and palmitic acid were relatively increased according to the oxidation proceeded. However, the concentrations of these fatty acids were hardly changed in samples stored at 5 °C. These changes were especially more notable in the lipids from RAP than those from PAP during storage. Little difference in fatty acid composition was noted between neutral lipids and triglycerides in the samples.

Key words: adlay lipid, fatty acid composition of cereal lipid, processing and storage effect of cereal lipid

Introduction

Adlay (Coix Lachryma-jobi Linne var. Mayuen (Roman) Stapf) belonged to Graminaceae was used for food and medicine and its powder is recently used as adlay tea. The lipid content of adlay represents a small fraction, but this lipid undoubtedly plays an important functional role in their products during processing and storage[1]. One of the major chemical reactions that take place during processing and storage is lipid deterioration caused by lipid oxidation[2]

During the storage of milled rice, the fatty acid compositions of total lipid did not change, but those of the free fatty acid fraction changed significantly[3]. At present, very little is known about the nature of fatty acid composition of the lipids in adlay during processing and storage. This study was performed to investigate the detailed changes of fatty acid composition in adlay lipids by the conditions of processing and storage. In this paper continuing previous paper[4], the changes of fatty acid composition in RAP and PAP stored at 5 °C and 35 °C for six months were reported.

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Materials and Methods

Preparation of adlay powder

The preparation method of raw adlay powder (RAP) and processed adlay powder (PAP) and storage conditions were treated as described in previous paper[4].

Lipid extraction and fractionation

Crude lipids were extracted with a mixture of chloroform-methanol (2:1, v/v) as described by Folch *et al.*[5] Crude lipid extracts were freed from non-lipid contaminants by applying a portion (200 mg) of the crude lipids to a column of sephadex G-25, and eluting the lipids with a mixture of water-saturated chloroform/methanol (19/1, v/v)[6].

The purified lipids were subjected to column chromatography, using a silicic acid (100 mesh, Mallinckrodt Chemical Works, USA) column[7]. Chloroform, aceton and methanol were used sequentially to elute the neutral lipid(NL), glycoli-

pid(GL) and phospholipid(PL), respectively. For separation of triglyceride(TG) and free fatty acid (FFA), preparative TLC on silica gel-G and a solvent system containing hexane-diethyl ether-acetic acid (80:20:1, v/v) were used. TG and FFA were located under ultraviolet light after spraying with Rhodamine-6G in methanol. The bands were scrapped off the plate and extracted with chloroform-methanol (2:1, v/v)[8].

Fatty acid analysis

Fatty acid esters were prepared by transesterification of the oil using a 5% solution of hydrochroric acid in methanol[9] and analysed using a gas chromatograph (Shimadzu GC-7AG, Japan) equipped with flame ionization detector. A glass column (3.1m × 3.2mm, i.d.) packed with 15% (w/w) DEGS on Shimalite W(60-80 mesh) was used for methyl ester seperation. The column oven temperature was 195 °C, the injection temperature was maintained at 250 °C and detector at 250 °C. Flow rate of the nitrogen carrier gas was 35 m*l* per min. The emerging peaks were identified by comparing retention time to those of a standard mixture of known fatty acid methyl esters. Peak areas were integrated and the percentage of total fatty acids were determined[10].

Results and Discussion

Fatty acid compositions of lipid fractions and their changes during storage

Table 1 and 2 show the fatty acid compositions of lipid fraction in RAP and PAP stored at 5 °C and 35 °C. Fig. 1 shows the relative changes of both oleic and linoleic acid in the neutral lipid fraction during storage at 5 °C and 35 °C.

The results show that oleic, linoleic and palmitic acid were the major fatty acids in each lipid fraction of RAP and PAP, while smaller quantities of stearic and linolenic acid were also detected. In NL fraction, oleic acid was the predominant unsaturated fatty acid and was present in the amount of 45-46% of total fatty acids, while in GL and PL fraction, linoleic acid was the predominant unsaturated fatty acid. The higher amounts of linoleic acid in GL and PL fraction may cause increased oxidative deterioration during processing and

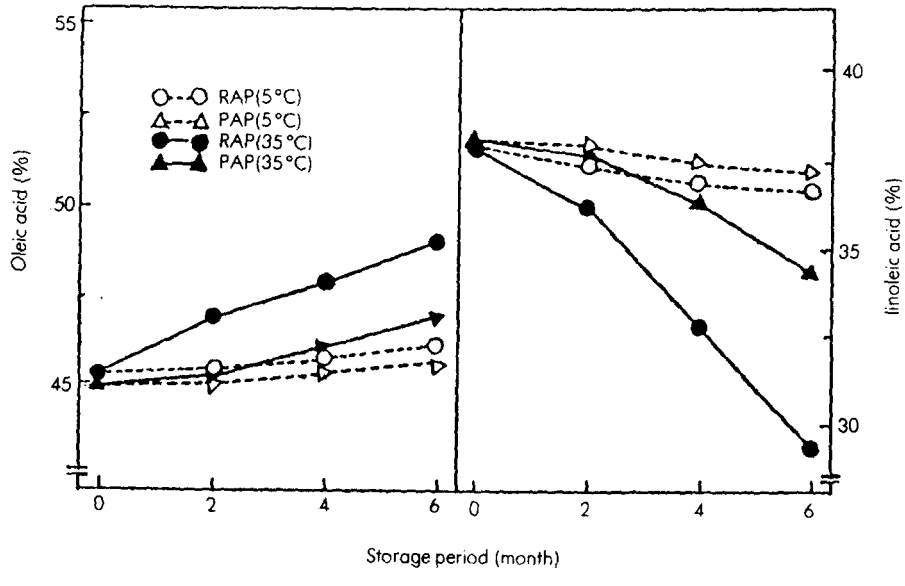

Fig. 1. Relative changes in the contents of oleic and linoleic acid of neutral lipid in raw adlay powder (RAP) and processed adlay powder (PAP) during storage at 5 °C and 35 °C.

Table 1. Changes in fatty acid composition of various lipid fraction extracted from raw adlay powder during storage at 5°C and 35°C (Area %)

Fatty acids	5°C											
	Neutral lipids				Glycolipids				Phospholipids			
	0	2	4	6[a]	0	2	4	6[a]	0	2	4	6[a]
14:0	0.1	0.2	0.0	0.1	0.2	0.2	0.2	1.1	0.2	0.2	0.7	0.9
16:0	14.1	14.3	15.0	15.7	18.3	18.8	19.3	19.0	17.4	18.4	18.5	18.6
18:0	1.3	1.5	1.1	0,7	2.5	2.1	2.2	3.0	0.9	0.9	0.7	0.5
18:1	45.2	45.2	45.8	46.1	27.9	27.6	28.1	29.0	40.1	40.1	40.6	41.1
18:2	37.9	37.5	36.8	36.7	50.2	50.1	47.3	46.2	40.4	39.0	38.3	38.2
18:3	1.2	1.1	1.2	0.5	0.7	0.7	2.0	1.1	0.6	0.7	0.8	0.5
20:1	0.2	0.2	0.1	0.2	0.2	0.5	0.9	0.6	0.4	0.7	0.4	0.2
SFA	15.5	16.0	16.1	16.5	21.0	21.1	21.7	23.1	18.5	19.5	19.9	20.0
MUFA	45.5	45.4	45.9	46.3	28.1	28.1	29.0	29.6	40.5	40.8	41.0	41.3
PUFA	39.1	38.6	38.0	37.2	50.9	50.8	49.3	47.3	41.0	39.7	39.1	38.7
S/M/P	0.4/1.2/1.0	0.4/1.2/1.0	0.4/1.2/1.0	0.4/1.2/1.0	0.4/0.6/1.0	0.4/0.6/1.0	0.4/0.6/1.0	0.5/0.6/1.0	0.5/1.0/1.0	0.5/1.0/1.0	0.5/1.0/1.0	0.5/1.1/1.0
P/S	2.5	2.4	2.4	2.3	2.4	2.4	2.3	2.0	2.2	2.0	2.0	1.9

Fatty acids	35°C											
	Neutral lipids				Glycolipids				Phospholipids			
	2	4	6[a]		2	4	6[a]		2	4	6[a]	
14:0	0.0	0.1	0.2		1.0	1.0	0.9		0.6	0.6	0.7	
16:0	15.0	16.1	18.7		19.9	22.3	24.3		19.6	22.0	23.8	
18:0	1.2	1.3	2.1		2.0	2.1	2.1		1.4	2.1	3.2	
18:1	47.0	48.2	49.1		29.0	32.4	35.5		36.6	28.7	24.2	
18:2	36.1	33.0	29.2		46.2	40.0	35.5		36.6	28.7	24.2	
18:3	0.5	0.8	0.5		0.8	1.2	0.9		1.0	1.6	2.1	
20:1	0.2	0.5	0.2		1.1	1.0	1.1		0.6	0.8	0.7	
SFA	16.2	17.5	21.0		22.9	25.4	27.3		21.6	24.7	27.7	
MUFA	47.2	48.7	49.3		30.1	33.4	36.3		40.8	45.0	46.0	
PUFA	36.6	33.8	29.7		47.0	41.2	36.4		37.6	30.3	26.3	
S/M/P	0.1/1.3/1.0	0.5/1.4/1.0	0.7/1.7/1.0		0.5/0.6/1.0	0.6/0.8/1.0	0.8/1.0/1.0		0.6/1.1/1.0	0.8/1.5/1.0	1.1/1.8/1.0	
P/S	2.3	1.9	1.4		2.1	1.6	1.3		1.7	1.2	1.0	

a) Values mean the storage period (month).

Abbreviation: SFA or S, saturated fatty acid; MUFA or M, monounsaturated fatty acid; PUFA or P, polyunsaturated fatty acid.

Table 2. Changes in fatty acid composition of various lipid fraction extracted from processed adlay powder during storage at 5°C and 35°C (Area %)

Fatty acids	5°C											
	Neutral lipids				Glycolipids				Phospholipids			
	0	2	4	6[a)]	0	2	4	6[a)]	0	2	4	6[a)]
14:0	0.1	0.1	0.1	0.0	0.1	0.2	0.2	0.9	0.4	0.4	0.4	0.4
16:0	14.4	14.8	14.9	15.5	18.4	19.0	19.3	19.3	17.5	18.0	18.1	18.3
18:0	1.2	1.2	1.3	1.2	1.9	1.8	1.9	1.8	1.8	1.4	1.6	1.8
18:1	45.1	45.2	45.3	45.3	32.0	32.9	33.0	33.0	37.0	37.5	37.5	37.5
18:2	38.0	37.8	37.2	37.2	45.9	43.6	43.5	43.4	41.0	40.7	40.1	39.6
18:3	1.0	0.8	0.6	0.5	1.4	1.7	1.6	1.3	1.9	1.7	1.6	1.5
20:1	0.2	0.1	0.6	0.3	0.3	0.8	0.5	0.3	0.4	0.3	0.7	0.9
SFA	15.7	16.1	16.3	16.7	20.4	21.0	21.4	22.0	19.7	19.8	20.1	20.5
MUFA	45.3	45.3	45.9	45.6	32.2	33.7	33.5	33.3	37.4	37.8	38.2	38.4
PUFA	39.0	38.6	37.8	37.7	47.3	45.3	45.1	44.7	42.9	42.4	41.7	41.4
S/M/P	0.4/1.2/1.0	0.4/1.2/1.0	0.4/1.2/1.0	0.4/1.2/1.0	0.4/0.7/1.0	0.5/0.7/1.0	0.5/0.7/1.0	0.5/0.7/1.0	0.5/0.9/1.0	0.5/0.9/1.0	0.5/0.9/1.0	0.5/0.9/1.0
P/S	2.5	2.4	2.3	2.3	2.3	2.2	2.1	2.0	2.2	2.1	2.1	2.0

Fatty acids	35°C									
	Neutral lipids			Glycolipids			Phospholipids			
	2	4	6[a)]	2	4	6[a)]	2	4	6[a)]	
14:0	0.1	0.1	0.1	0.3	0.4	0.8	0.4	0.4	0.2	
16:0	15.0	16.0	17.1	19.8	22.2	23.7	17.8	19.6	21.7	
18:0	1.0	0.9	0.8	1.8	1.9	2.4	1.2	1.8	1.8	
18:1	45.2	46.0	46.8	33.7	34.1	34.6	37.6	38.0	38.3	
18:2	37.7	36.0	34.2	42.0	39.1	36.4	41.0	38.0	36.1	
18:3	0.8	0.7	0.6	1.6	1.5	1.6	1.7	1.4	1.4	
20:1	0.2	0.3	0.4	0.8	0.8	0.5	0.3	0.8	0.5	
SFA	16.1	17.0	18.0	21.9	24.5	26.9	19.4	21.8	23.7	
MUFA	45.4	16.3	47.2	34.5	34.9	35.1	37.9	38.8	38.8	
PUFA	38.5	36.7	34.8	43.6	40.6	38.0	42.7	39.4	37.5	
S/M/P	0.4/1.2/1.0	0.5/1.3/1.0	0.5/1.4/1.0	0.5/0.8/1.0	0.6/0.9/1.0	0.7/0.9/1.0	0.5/0.9/1.0	0.6/1.0/1.0	0.6/1.0/1.0	
P/S	2.4	2.2	1.9	2.0	1.7	1.4	2.2	1.8	1.6	

a) Values mean the storage period (month).

Abbreviation: SFA or S, saturated fatty acid; MUFA or M, mbnounsaturated fatty acid: PUFA or P, polyunsaturated fatty acid.

storage. The fatty acid compositions of RAP and PAP were similar to those studied on other cereal such as barley and brown rice[1].

Throughout the storage period, the changes in the fatty acid composition of each lipid fraction depended on the storage temperature and period. In NL fraction, the percentages of linoleic acid decreased, while those of oleic and palmitic acid relatively increased. On the other hand, the patterns of change in the fatty acid composition of GL were similar to the NL. During storage, the percentage of linoleic acid decreased and those of oleic and palmitic acid relatively increased. The patterns of change were more marked in GL containing the more amount of linoleic acid than NL. The patterns of change in fatty acid composition of PL were similar to the GL. Oleic and palmitic acid in PL tended to increase slightly, whereas linoleic acid relatively decreased during the storage period. These changes were especially significant at higher temperature (35 °C).

Changes of fatty acid composition in RAP and PAP during storage

The PAP had lower linoleic acid and higher oleic acid than the RAP at the initial stage. The decrease of linoleic acid in PAP may have been due to the heat treatment during the processing. The changes in the fatty acid composition of each lipid fraction in RAP and PAP depended on the storage temperature and period. In NL fraction, the percentage of linoleic acid decreased while those of oleic and palmitic acid relatively increased, especially markedly in the RAP stored at 35 °C. Lipoxygenase, which is principally in a plant enzyme, is known to be distributed widely in cereals and legumes and catalysed the peroxidation of polyunsaturated fatty acids containing cis, cis-1,4-pentadiene moieties by molecular oxygen[11]. The most common substrate for lipoxygenase in food materials is linoleic acid. Therefore, the greater decrease of linoleic acid in the raw sample may have been due to the fact that both autoxidation and lipoxygenase catalysed oxidation were occurring. In GL and PL fraction containing the more amount of linoleic acid, the RAP had a higher rate of degradation of linoleic acid than PAP. These patterns were markedly changed at higher temperature (35 °C), but were hardly changed during storage at 5 °C. This may

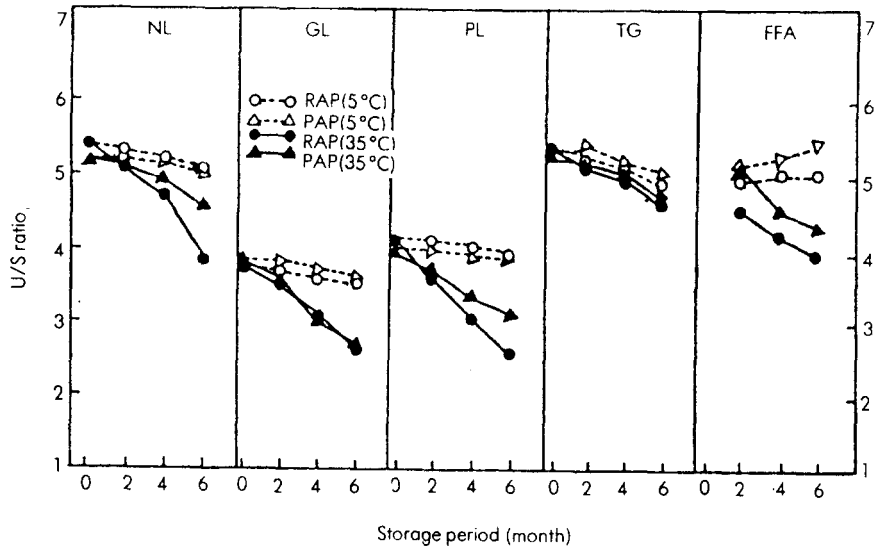

Fig. 2. Effects of temperature and storage time on the unsaturated/saturated ratio of fatty acid in various lipid fractions extracted from raw adlay powder (RAP) and processed adlay powder (PAP).
Abbreviations: NL; neutral lipid, GL; glycolipid, PL; phospholipid, TG; triglyceride, FFA; free fatty acid

Table 3. Changes in fatty acid composition of triglyceride in raw adlay powder (RAP) and processed adlay powder (PAP) during storage at 5°C and 35°C (Area %)

Fatty acids	5°C							
	RAP				PAP			
	0	2	4	6[a)]	0	2	4	6[a)]
14:0	0.4	0.2	0.3	0.1	0.3	0.2	0.2	0.2
16:0	13.9	14.1	14.5	15.8	14.0	14.0	14.2	15.3
18:0	1.2	1.5	1.2	0.8	1.2	1.5	1.7	1.0
18:1	49.4	49.4	49.4	49.5	48.0	48.0	48.1	48.3
18:2	34.3	34.1	34.0	33.1	35.2	35.1	34.5	34.1
18:3	0.6	0.5	0.4	0.4	1.1	1.0	1.1	0.9
20:1	0.2	0.2	0.2	0.3	0.2	0.2	0.2	0.2
SFA	15.5	15.8	16.0	16.7	15.5	15.7	16.1	16.5
MUFA	49.6	49.6	49.6	49.8	48.2	48.2	48.3	48.5
PUFA	34.9	34.6	34.4	33.5	36.3	36.1	35.6	35.0
S/M/P	0.4/1.4/1.0	0.5/1.4/1.0	0.5/1.4/1.0	0.5/1.5/1.0	0.4/1.3/1.0	0.4/1.3/1.0	0.5/1.4/1.0	0.5/1.4/1.0
P/S	2.3	2.2	2.2	2.0	2.3	2.3	2.2	2.1

Fatty acids	35°C							
	RAP				PAP			
	0	2	4	6[a)]	0	2	4	6[a)]
14:0	0.4	0.1	0.2	0.2	0.3	0.1	0.7	0.2
16:0	13.9	14.6	15.0	15.3	14.0	14.0	14.4	15.5
18:0	1.2	1.3	1.5	2.0	1.2	1.4	1.5	1.6
18:1	49.4	49.5	49.8	50.3	48.0	48.0	48.6	49.3
18:2	34.3	33.1	32.2	30.5	35.2	34.8	33.2	32.1
18:3	0.6	0.5	0.4	0.9	1.1	1.5	1.5	1.0
20:1	0.2	0.9	0.9	0.8	0.2	0.2	0.1	0.3
SFA	15.5	16.0	16.7	17.5	15.5	15.5	16.6	17.3
MUFA	49.6	50.4	50.7	51.1	48.2	48.2	48.7	49.6
PUFA	34.9	33.6	32.6	31.4	36.3	36.3	34.7	33.1
S/M/P	0.4/1.4/1.0	0.5/1.5/1.0	0.5/1.6/1.0	0.6/1.6/1.0	0.4/1.3/1.0	0.4/1.3/1.0	0.5/1.4/1.0	1.5/1.4/1.0
P/S	2.3	2.1	2.0	1.8	2.3	2.3	2.1	1.9

a) Values mean the storage period (month).

Abbreviation: SFA or S, saturated fatty acid; MUFA or M, monounsaturated fatty acid; PUFA or P, polyunsaturated fatty acid.

have been due to the more active action of lipoxygenase during storage at 35°C.

Changes in the ratio of unsaturated fatty acid to saturated fatty acid

Changes in the ratio of unsaturated to saturated fatty acid (U/S ratio) at various stage of this experiment are shown in Fig. 2. The changes in U/S ratio were used as a measure of changes in lipid composition of stored products. Since lipid oxidation is associated almost exclusively with unsaturated fatty acids, it can be expected that the U/S ratio decreases as a result of lipid oxidation during storage. The raw sample had higher percentage change of U/S ratio than processed sample. Again, a combination of lipoxygenase cata-

Table 4. Changes in fatty acid composition of free fatty acid in raw adlay powder (RAP) and processed adlay powder (PAP) during storage at 5°C and 35°C. (Area %)

Fatty acids	5°C							
	RAP				PAP			
	0	2	4	6[a]	0	2	4	6[a]
14:0	0.1	0.1	0.5	0.3	0.2	0.2	0.2	0.2
16:0	15.7	15.6	15.5	15.7	14.3	14.3	14.2	14.1
18:0	0.8	0.9	0.4	0.5	1.7	1.7	1.6	1.1
18:1	41.1	41.1	41.2	41.3	42.5	42.5	42.6	42.8
18:2	40.9	40.7	40.8	40.8	39.7	39.8	40.0	40.2
18:3	0.9	0.9	0.8	0.7	0.9	0.8	0.5	0.8
20:1	0.7	0.7	0.8	0.7	0.7	0.7	0.9	0.8
SFA	16.6	16.6	16.4	16.5	16.2	16.2	16.0	15.4
MUFA	41.8	41.8	42.0	42.0	43.2	43.2	43.5	43.6
PUFA	41.8	41.6	41.6	41.5	40.6	40.6	40.5	41.0
S/M/P	0.4/1.0/1.0	0.4/1.0/1.0	0.4/1.0/1.0	0.4/1.0/1.0	0.4/1.1/1.0	0.4/1.1/1.0	0.4/1.1/1.0	0.4/1.1/1.0
P/S	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.7

Fatty acids	35°C							
	RAP				PAP			
	0	2	4	6[a]	0	2	4	6[a]
14:0	0.1	0.2	0.4	0.3	0.2	0.1	0.5	0.8
16:0	15.7	16.1	16.8	17.8	14.3	14.2	16.0	16.4
18:0	0.8	1.6	1.8	2.1	1.7	1.3	1.3	1.4
18:1	41.1	40.8	41.4	42.8	42.5	42.4	42.8	43.2
18:2	40.9	40.3	38.4	36.5	39.7	40.4	38.1	37.0
18:3	0.9	0.8	0.7	0.3	0.9	0.9	0.3	0.5
20:1	0.7	0.2	0.5	0.2	0.7	0.7	1.0	0.7
SFA	16.6	17.9	19.0	20.2	16.2	15.6	17.8	18.6
MUFA	41.8	41.0	41.9	43.0	43.2	43.1	43.8	43.9
PUFA	41.8	41.1	39.1	36.8	40.6	41.3	38.4	37.5
S/M/P	0.4/1.0/1.0	0.4/1.0/1.0	0.5/1.1/1.0	0.6/1.2/1.0	0.4/1.1/1.0	0.4/1.0/1.0	0.5/1.1/1.0	0.5/1.2/1.0
P/S	2.5	2.3	2.1	1.8	2.5	2.6	2.2	2.0

a) Values mean the storage period (month).

Abbreviation: SFA or S, saturated fatty acid; MUFA or M, monounsaturated fatty acid; PUFA or P, polyunsaturated fatty acid

lysed autoxidation and autoxidation is inferred. In this study, changes of U/S ratio during adlay storage were lower than those of dried soybean curds during storage. It may be due to the fact that soybean oil has a higher linolenic acid content than adlay[12].

The changes of fatty acid composition in triglyceride and free fatty acid during storage

Table 3 and 4 show the fatty acid composition of triglyceride(TG) and free fatty acid (FFA) in RAP and PAP stored at 5°C and 35°C. Fatty acid compositions of TG were similar to the NL due to the fact that TG constituted of the majority of NL.

As the storage period went by, the percentages of linoleic acid decreased and those of palmitic and

oleic acid relatively increased. After four months, this patterns of change were distinct and especially significant in RAP stored at higher temperature (35 °C). It was clear that the percentage of increase in oleic acid of FFA fraction was roughly accounted for by the amount of decrease in corresponding fatty acid of TG and each lipid fraction. This results implied that the decreased amount of oleic acid in TG and each lipid fraction seemed to be directly transferred to the FFA fraction. However, the decreased amount of linoleic acid in the TG and each lipid fraction was greater than its increased amount in the FFA fraction at storage temperature of 35 °C. This means that a part of the linoleic acid from the TG and each lipid fraction was converted to oxidized compounds. This pattern was more marked in RAP by the action of lipoxygenase. This seemed to agree to the state occurring in brown rice stored at high temperature (35 °C). FFA in rice were released from the neutral lipid during storage and the increase in their amounts resulted in the deterioration of rice flavor[13].

FFA was decomposed differently according to fatty acid composition. Methyl linoleate reacted 10-40 times quicker than oleate because of the enhanced reactivity of the C-11 methylene group lying between the double bonds[14]. Therefore, oxidation of oleic acid was a slow reaction occurring only after a long induction period and more stable than that of linoleic acid. The FFA of RAP contained less linoleic acid and more oleic acid than those of PAP during storage, their degree being much more striking when the storage temperature was at 35 °C. This may have been due to the more active action of lipoxygenase during storage at 35 °C. The FFA of old rice contained less palmitic, linoleic and linolenic acid and more oleic acid than those of fresh rice[15].

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저장중 율무가루 지방질의 지방산 조성의 변화

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생 율무가루와 가공(침지, 증자, 건조)된 율무가루를 5℃ 및 35℃에서 6개월 동안 저장하면서 지방질에 있는 지방산 조성의 변화를 조사하였다. 각 획분별 지방질의 주요지방산은 oleic, linoleic, palmitic acid였으며, stearic 및 linolenic acid도 소량으로 함유되어 있었다. 35℃에서 저장하는 동안에 linoleic acid의 조성비는 감소하였으며 상대적으로 oleic 및 palmitic acid의 조성비는 증가하였으나, 5℃에서는 거의 변화가 없었으며, 생 율무가루가 가공된 율무가루보다도 더욱 현저하게 변화하였다. TG에 함유된 지방산 조성의 변화는 중성지방질과 거의 유사하였으며, 유리지방산은 35℃에서 저장한 것이 5℃에서 저장한 것보다 oleic acid를 많이 함유한 반면, linoleic acid를 적게 함유하였으며 이러한 현상은 생 시료에서 더 현저하였다.