DETERMINATION OF FREE FATTY ACID IN SOYBEAN

OIL BY TITRATION FROM FOOD AND DRUGS

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TECHNICAL REPORT

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ABSTRACT

AOCS official method Ca 5a-40 for determination of free fatty acids (FFA) was used to determine FFA in soybean oils. The method used about 2.0 g of the weight of oil sample; alcohol volume was 50 ml and the alkali strength of 0.1 N NaOH as recommended in the official method. Standard solutions of the soybean oil were prepared by weighing 2.0 g of the soybean oil and adding 50 ml of ethanol. The solution was heated and was titrated with 0.1 N NaOH. The FFA concentrations were then determined by calculations.

CHAPTER 1

1.0 Introduction

1.1 Degradation and Rancidity

Oils and fats commence to decompose from the moment they are isolated from their natural living environment. The presence of free fatty acids is an indication of lipase activity or other hydrolytic action. Changes occur during storage that results in the production of an unpleasant taste and odour. Such oils and fats are referred to as having become rancid. The unpleasant organoleptic characteristics are in part caused by the presence of free fatty acids but the major development of rancidity is brought about by atmospheric oxidation (auto oxidation). Oxidative rancidity accelerated by exposure to heat and light, by moisture and by the presence of traces of transition metals e.g. copper, nickel, iron and residual natural dyes and pigments (Kirk and Sawyer, 1991). Oxygen is taken up by the fat with the formation of hydroperoxides (ROOH). These are usually referred to as peroxides. The presence of natural antioxidants e.g. tocopherols or synthetic antioxidants inhibit the formation of peroxides. The initial oxidation of fat is usually slow and is at a relatively uniform rate. This is known as induction period. With most oils and fats, the free acidity increases during storage (Kirk and Sawyer, 1991).

CHAPTER 2

2.0 Literature Review

Free fatty acid (FFA) content is one of the most frequently determined quality indices during edible oils production, storage and marketing. It measures the extent to which hydrolysis has liberated fatty acids from their ester linkage with the parent triglyceride molecule (Teoh, 2006). Edible oils undergo various processing steps, resulting in low FFA content (Tandy et al, 1984). Accordingly, several papers have been published for the determination of FFA in biological fluids (Husek et al, 2002; Miwa, 2002; milk Shahin et al, 1987); fruit juices (Jakmunee et al, 2005); and fats and oils (Canhan and Pacey, 1987; Ekstrom, 1981; Ismail et al, 1993; Kwon and Rhee, 1986).

In the soybean industry, the presence and development of any amount of free fatty acid in soybean oil is undesirable. The FFA content of oil in soybeans indicates how well the beans have been treated during the period between harvest and processing. The eventual consequence of high FFA content is monetary loss because processing soybeans with high FFA content results in more refining loss. Norris (1982) estimates the potential loss at about three times the FFA content. Due to the economic impact of high FFA, it is important to determine the level of FFA in soybean oil and also monitor its concentration during processing.

The standard method for FFA determination in extracted crude and refined soybean oil samples is based on acid-base titration technique in a non-aqueous system. The method commonly used is AOCS official method 5a-40. A simple and inexpensive method is needed that accurately quantifies FFA content in soybean oil. The use of smaller amounts of chemicals shall be useful, especially in developing countries, where chemicals are scarce and expensive.

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Also, it is reasonable to limit the use of resources, those particularly hazardous to the environment. A smaller size sample shall also allow savings of storage space in laboratories that handle large numbers of samples and if protocol urges that soybean oil samples be kept after analysis.

Lanser et al, (1991) developed a user-interactive computer-assisted Fourier transform infrared (FTIR) method to estimate FFA content in soybean oil samples. The method requires only one drop of soybean oil. The limitation of the method and other similar spectophotometric methods such as that described by Canham and Pacey, 1987, is the cost of instruments: it is prohibitive in most laboratories. Lanser et al, 1991 also evaluated a modified version of the official method in which between 6 and 7 g of oil is used. The coefficient of determination (R^2) of the modified method was 0.999. The high correlation indicates that the officially recommended sample size can be scaled down without losing accuracy of FFA determination.

The objective of this research was to determine FFA content in soybean oil by acid-base titration method by using an oil sample of about 10 % (between 1-10 g) of the weight recommended in the AOCS official method.

CHAPTER 3

3.0 Materials and Method

Soybean oil samples were obtained from Food and Drug Authority, Ghana. AOCS official method Ca 5a-40. FFA content of soybean oil is usually determined by titration with a standard alkali, either 0.1 M NaOH or 0.1 M KOH of specific strength or normality. For AOCS official method Ca 5a-40 (1), the recommended oil sample size, volume of alcohol and NaOH strength are 1-10 g of oil, 50-100 ml and 0.1 N respectively. FFA concentration in fats and oils is calculated as percentage oleic acid. The expression as given in AOCS official method Ca 5a-40 (1) is,

$$\% \text{ FFA as oleic acid} = \frac{alkali\ volume\ (ml)\ x\ alkali\ normality\ x\ 28.2}{sample\ weight\ (g)}$$

Preparation of Standard Soybean Oil for Titration

About 2.0 g of oil samples each in duplicates were transferred into Erlenmeyer flask. Then, 50 ml of ethanol was added and was heated on a hot plate with stirring. The oil containing the heated ethanol was titrated with 0.1 N NaOH or 0.1 N KOH by addition of 2-3 drops phenolphthalein as indicator. Weighing of the oil samples and titrations were carried out in duplicates as shown in Table 3.1.

Table 3.1 Sample size and reagent concentrations used in the methods

Soybean oil Sample No.	Weight of soybean Sample (g)	Vol. of ethanol (ml)	Normality of NaOH (N)
S$_2$	2.0313	50	0.1
	2.0491		
S$_3$	2.0437	50	0.1
	2.0204		
S$_4$	2.0481	50	0.1
	2.0259		
S$_5$	2.0479	50	0.1
	2.0392		

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S_6	2.0490	50	0.1
	2.0684		
S_7	2.0151	50	0.1
	2.0386		
S_8	2.0526	50	0.1
	2.0572		
	2.0168	50	0.1
	2.0600		
S_9	2.0388	50	0.1
	2.0296		
S_{10}	2.0731	50	0.1
	2.0409		
S_{11}	2.0655	50	0.1
	2.0430		
S_{12}	2.0654	50	0.1
	2.0537		
S_{13}	2.0897	50	0.1
	2.0629		
S_{14}	2.0656	50	0.1
	2.0672		
S_{15}	2.0105	50	0.1
	2.0899		
S_{16}	2.0495	50	0.1
	2.0185		
S_{17}	2.0369	50	0.1
	2.0035		
S_{18}	2.0654	50	0.1
	2.0537		
S_{19A}	2.0101	50	0.1
	2.0382		
S_{20}	2.0313	50	0.1
	2.0491		
S_{21}	2.0104	50	0.1
	2.0383		
S_{22}	2.0409	50	0.1
	2.0625		
1_A	2.0654	50	0.1
	2.0537		
1_B	2.0341	50	0.1
	2.0432		
2	2.0331	50	0.1
	2.0654		

CHAPTER 4

4.0 Results and Discussion

Titration method: Preliminary determination FFA in the soybean oil samples showed that the NaOH concentration needed to detect and quantity FFA content was about 0.1001 N. Based on this estimation, NaOH concentrations for the titration method was kept constant. Alcohol volume used in this method was also kept constant as that used in the AOCS official method (Table 3.2). Table values served only as guidelines in preparing samples and reagents. The highest % FFA was found in soybean oil S13 with FFA value of 1.7 %. The lowest value of FFA was found in soybean oil S4 and soybean oil S10 with FFA values of 0.1 %.

The soybean oil S2 had FFA of 0.6 % while S3 registered 0.3 %. S4, S5 and S6 recorded 0.1 %, 0.2 % and 0.5 % respectively: With S7, S8 and S9, recorded 0.3 %, 0.6 % and 0.6 % respectively. S10, S11 and S12 had 0.1%, 1.6% and 0.35. S12, S13 and S14 gave 0.3%, 1.7% and 0.6 % FFA. S15, S15 and S17 also yielded 0.7 %, 0.6 % and 0.6 % FFA. S18, S19A and S19B also gave 0.3 %, 0.7 % and 0.2 % FFA. S20, S21 and S22 also produced 0.3 %, 0.5 % and 0.4 % FFA. Soybean oil samples 1A and 1B also registered 1 % and 0.5 % FFA respectively.

Conclusion

Acid-base titration method is the most simple and inexpensive method for determination of FFA in soybean oils. It also saves time. The method is good for those of us in the developing countries where chemicals are scarce and expensive. It is also reasonable to limit the use of chemicals, especially those hazardous to the environment.

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Appendix

Results of FFA in soybean oil from Food and Drugs Authority (FDA).

Soybean oil samples	% FFA (%)
S3	0.3
S4	0.1
S5	0.2
S6	0.5
S7	0.3
S8	0.6
S9	0.6
S10	0.1
S11	1.6
S12	0.3
S13	1.7
S14	0.6
S15	0.7
S16	0.6
S17	0.6
S18	0.3
S19A	0.7
S19B	0.2
S20	0.3
S21	0.5
S22	0.4
1A	1
1B	0.5