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Quick Test for Quality Deterioration in Processed Foods

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ABSTRACT

A quick test for quality deterioration of milk powder, refined oil and *atta*, based on the indicator test solution was studied. The indicator test solution comprising *NaOH* and bromothymol blue indicator remains stable for 6 months in glass bottles. Depending on the acidity of the sample, the colour changed from blue to light blue to green to yellow. The concentration of the indicator test solution is optimised for each product, viz., 4 ml for milk powder and refined oil and 2 ml for *atta*, to which 1 ml aqueous sample or oil is added which gives the colour change. The appearance of green colour is considered the cutoff point for inferior quality and is based on the limits specified by the Bureau of Indian Standards for these products. The test has proved its efficacy in quality checking during storage of milk powder and deep fat frying of products in refined oils.

1. INTRODUCTION

Processed foods such as milk powder, *atta*, and refined oils are being purchased in bulk by the Armed Forces. The long-distance transportation from the supply depots to the consumer units takes a lot of time, and also transportational hazards sometimes call for checking the quality of food before consumption. The quick test for checking the quality of processed foods is desirable.

During the storage of milk powder, hydrolytic reactions are predominant resulting in increase in acidity. Besides, browning reactions and thermal abuse reduce the solubility of milk powder. During the storage of *atta* the insect infestation and increased acid value are some of the changes found normally. Though the storage deterioration of refined vegetable oils leads to autoxidation, forming peroxides, aldehydes, and ketones, some hydrolytic reactions also occur but at a slow rate, leading to the formation of free fatty acids. However, improper refining and frying operations indicate increased acid value, and thus, acid value can be taken as an index for checking the oil quality. Fritsch,[1] *et al.* utilised dielectric constant of an oil to examine its deterioration. Generally titrimetric methods are used for determining the acid value. The quick test[2] reported is also based on titrimetric analysis which requires certain laboratory facilities. In the present study, a simple, quick test based on the indicator test solution has been developed which can be used for some of the processed products where acidity is regarded as one of the quality parameters.

2. MATERIALS & METHODS

Reagents and chemicals used were of AR grade. The glass test tubes were marked suitably (1,2,4,5,10 ml) to conduct the test. Milk powder and refined oils were procured locally with the latest date of manufacture.

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2.1 Preparation of Indicator Test Solution

To *NaOH* (1 g) dissolved in water, polyvinyl alcohol (1 g) was added and heated for 30 min at about 90 °C. To this solution, Triton 100 (0.2 g) was added and heating continued to obtain clear transparent solution. Bromothymol blue indicator (0.1 g) was added to the cooled solution and shaken. This served as a stock solution. Stock solution (2 ml) was diluted to 100 ml with C_2H_5OH to serve as the indicator test solution[3] used for checking the extent of deterioration in foods.

2.2 Preparation of Sample for Test

2.2.1 Milk Powder

To 1 ml, 10 ml marked tubes, milk powder was transferred up to first mark, followed by addition of distilled water to the second mark and shaken well. To 4 ml, 5 ml marked tubes, indicator test solution was transferred up to the first mark, followed by careful addition of milk powder solution (to be tested) up to the second mark and shaken well. The colour change gives the extent of acidity.

2.2.2 Refined Oils

To 4 ml, 5 ml marked tubes, indicator test solution was transferred up to the first mark, followed by careful addition of oil (to be tested) up to the second mark and shaken well. The colour change gives the extent of acidity.

2.2.3 Atta

To 1 ml, 10 ml marked tubes, test sample of *atta* was transferred up to the first mark, followed by distilled water up to the second mark and shaken well. To 4 ml, 5 ml marked tubes, indicator test solution was transferred up to the first mark, followed by aqueous extract of *atta* up to the second mark and shaken well. The colour change gives the extent of acidity.

The titratable acidity in milk powder[4], *atta*[5] and free fatty acids in oils[6] were determined using standard procedures. Browning in milk powder was determined by extracting the sample (5 g) with 50 ml of 70 per cent alcohol by shaking in a mechanical shaker, followed by filtration. The absorbance of the filtrate was read at 420 nm. The peroxide value in milk powder was determiend by American Oil Chemists Society (AOCS) method[7]. To evaluate the reconstitution characteristics, the milk powder samples during storage were reconstituted in warm water in 1:5 ratio and shaken well.

3. RESULTS & DISCUSSION

Simple tests with readymade indicator test solution and the sample without much preparation for testing would be convenient to assess the quality of the products. With this view, the acid-base indicator with a predetermined quantity of alkali in a solution was used for developing the spot test for acidity determination. The acid-base indicators with varied *p*H range, such as bromocresol purple (5.2-6.8), methyl red (4.2-6.3), bromothymol blue (6-7.6), bromophenol red (5.2-6.8) which change colour at moderate *p*H ranges were chosen. Among these, bromothymol blue which gave a distinct and gradational colour change from blue to light blue to green to yellow was most suitable, whereas other indicators changed colour at extreme limit *p*H range. However, AOAC method[8] refers to the use of bromothymol blue for colorimetric determination of *p*H, where *p*H is temperature dependent. *NaOH* and Na_2CO_3 solutions were used as alkali for the test. Though both the compounds showed similar results initially, *NaOH* was preferred. The decrease in colour was observed with Na_2CO_3 solution. Accordingly, the indicator test solution with predetermined *NaOH* and bromothymol blue indicator, along with other two additives was prepared and this had 6-months stability in glass bottle. Polyvinyl alcohol and triton 100, a higher alcohol used, impart protection during storage and also served as better media for dispersion of less, nonaqueous sample with the indicator solution. The quantity of the solution required for milk powder, *atta* and refined oil has been optimised, keeping in view the acidity limits as per Bureau of Indian Standards (BIS) specifications. Depending

Table 1. Colour test for milk powder samples

Commercial brands (Code No.)	Acidity by titrimetry (%)	Colour with indicator test solution
A_1	0.52	Blue
A_2	0.87	Blue
B	0.99	Blue
C_1	1.01	Blue
D_1	1.04	Blue
E_1	1.05	Blue
F_1	1.15	Light-blue
G	1.16	Light-blue
C_2	1.35	Light-blue
H	1.45	Light-blue
D_2	1.53	Greenish-blue
F_2	1.70	Green
E_2	1.72	Green
I_1	1.72	Green
1_2	1.84	Green
I_3	1.91	Green
D_3	1.94	Green
E_3	1.91	Green

on the acidity of the sample, the colour changed from blue to light blue to green to yellow. Green is considered as the cutoff point for nonacceptability. As per BIS, the acidity limits for milk powder, refined oils and *atta* are 1.5 per cent, 0.25 per cent and 0.1 per cent, respectively. Accordingly, the dilution of the sample and the volume of the indicator test solution are optimised. It is 4 ml for milk powder and oils and 2 ml for *atta* samples. Different brands of milk powder were tested using the indicator test solution, and acidity was determined by BIS methods for comparison. The results are presented in Table 1. It may be observed that up to 1.05 per cent acidity, the colour remains blue and changes to light blue up to 1.5 per cent acidity, and above that the solution turns green. The samples which resulted in green colour had the higher acidity than the specified limits of 1.5 per cent and one sample with 1.53 per cent acidity showed greenish blue, which is on the higher side of the limit indicating the reliability of the test. Bromothymol blue test is used for finding *p*H of milk and to know whether milk is drawn from infected udder [9]. However, the bromothymol blue test is carried out by preparing fresh solution. Refined vegetable oils were tested using the

Table 2. Colour test for refined oils

Refined oil samples	Acidity by titrimetry (%)	Colour with indicator test solution
Sunflower oil	0.067	Blue
Sunflower oil	0.074	Blue
Sunflower oil	0.087	Blue
Cottonseed oil	0.092	Blue
Cottonseed oil	0.097	Blue
Cottonseed oil	0.099	Blue
Groundnut oil	0.100	Blue
Hydrogenated fat	0.100	Blue
Cottonseed oil	0.100	Blue
Sunflower oil	0.100	Blue
Cottonseed oil	0.113	Blue
Sunflower oil	0.115	Blue
Mustard oil	0.124	Blue
Sunflower oil	0.127	Blue
Cottonseed oil	0.131	Blue
Groundnut oil	0.150	Blue
Sunflower oil	0.150	Blue
Cottonseed oil	0.152	Blue
Groundnut oil	0.166	Light-blue
Groundnut oil	0.175	Light-blue
Mustard oil	0.190	Light-blue
Sunflower oil	0.192	Light-blue
Sunflower oil	0.210	Light-blue
Cottonseed oil	0.220	Light-blue
Groundnut oil	0.223	Light-blue
Groundnut oil	0.240	Light-blue
Groundnut oil	0.262	Green
Mustard oil	0.290	Green
Groundnut oil	0.290	Green
Mustard oil	0.310	Green
Mustard oil	0.380	Green
Groundnut oil	0.440	Greenish-yellow
Mustard oil	0.610	Yellow
Coconut oil	0.170	Light-blue
Coconut oil	0.27	Green
Coconut oil	0.38	Yellow

indicator test solution and the results are presented in Table 2. The indicator test solution remained blue up to 0.152 per cent free fatty acids and changed to light blue up to 0.24 per cent and developed green colour above 0.262 per cent and turned yellow with high free fatty acid value (> 0.45 per cent). The limit of 0.25 per cent of free fatty acids is specified as per Prevention of Food Adulteration Act (PFA) and the appearance of green colour is the cutoff point for checking the quality of oil. This is useful for screening the samples during

Table 3. Colour test for *atta* samples

Sample	Titratable acidity (%)	Colour with indicator solution
Atta	0.098	Blue
Atta	0.100	Blue
Atta	0.126	Light-blue
Atta	0.154	Bluish-green
Atta	0.180	Green
Atta	0.280	Yellow
Atta	0.330	Yellow
Chapati	0.150	Bluish-green
Chapati	0.150	Bluish-green
Chapati	0.180	Green

procurement. The results of the indicator test solution with *atta* and *chapati* are presented in Table 3. The maximum limit of alcoholic acidity for *atta* is 0.1 per cent and freshly ground *atta* is about 0.12 per cent, which reflects on the stored wheat itself. In case of *atta*, the change in indicator colour can be taken as index for nonacceptance. However, the method is applicable as per the results.

These quick tests will be applicable at the consumer units to check whether expected shelf life can be extended. With this view, the efficacy of the indicator test solution was tested during storage of milk powder samples packed in paper-*Al* foil-polyethylene laminate pouches. The results are presented in Table 4. It was observed that acidity increased with increase in storage period, and the colour test was at par with the observed value. Out of three commercial brands studied, two brands had acidity within the prescribed limits even after 12 months and the reconstituted product was acceptable. But one of the brands showed increased acidity, well reflected by the indicator test solution also. The other quality checking reactions that are expected in milk powder are the non-enzymatic browning and autoxidation of lipids. Accordingly, the samples were also analysed for browning which had remained almost the same throughout the storage period in two brands, and a considerable increase in browning was reflected in the third brand. Poor reconstitution of milk powder and the dark colour indicated nonacceptance of the sample just after a month. The rate of formation of peroxides was slow in the milk powder samples, indicating that acidity and browning are important parameters of concern. The colour test has shown good results during storage of milk powder.

The efficacy of the test solution during frying operations has been checked by testing the oils drawn from periodical frying of *poories*/potato chips up to 18 hr. The results are presented in Table 5. Cottonseed oil, groundnut oil and mustard oil were used as the frying medium. The free fatty acids increased with the time of frying and were well indicated by the concomitant colour developed with the test solution. The increase in free fatty

Table 4. Changes in quality parameters during storage of milk powder at 37 °C

Commercial brand milk powder	Storage period (months)	Acidity (%)	Colour with indicator test solution	Reconstitution	Browning (OD at 420 nm)	Peroxide value (meq O_2 / kg sample)
	0	0.68	Blue	Good	0.05	2.10
	4	0.87	Blue	Good	0.06	3.30
A	8	0.94	Blue	Good	0.07	4.10
	12	1.04	Blue	Good	0.07	4.10
	0	0.76	Blue	Good	0.06	1.93
B	4	0.81	Blue	Good	0.07	3.60
	8	0.88	Blue	Good	0.07	3.40
	12	1.10	Light-blue	Good	0.08	3.80
	0	1.23	Light-blue	Good	0.07	2.78
C	4	2.29	Yellow	Poor	1.12	6.25
	8	2.84	Yellow	Poor	1.71	6.80

Table 5. Changes in acidity and colour with indicator test solution during frying operations

Oil type	Duration of fraying (hr)	Free fatty acids (%)	Colour with indicator test solution
Cottonseed oil	0	0.092	Blue
	3	0.097	Blue
	6	0.110	Blue
	9	0.113	Blue
	12	0.131	Blue
	15	0.152	Blue
	18	0.220	Blue
Mustard oil	0	0.260	Green
	3	0.480	Green
	6	0.540	Yellow
	9	0.645	Yellow
	12	0.707	Yellow
	15	0.781	Yellow
	18	0.803	Yellow
Groundnut oil	0	0.150	Blue
	3	0.166	Blue
	6	0.175	Blue
	9	0.223	Blue
	12	0.241	Light-blue

acids during frying operations has also been reported earlier [10-13]

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