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ScienceDirect
Food Chemistry 106 (2008) 634–639
Food Chemistry
www.elsevier.com/locate/foodchem

Effect of fish (*Catla catla*) frying on the quality characteristics of sunflower oil

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Received 26 March 2007; accepted 20 June 2007

Abstract

The quality of sunflower oil, as affected by fish (*Catla catla*) frying at 180 °C for 14 h was investigated. The purpose of the study was to find out the quality deterioration of sunflower oil with respect to time as affected by fish frying and to generate equations that can be used for predicting the quality parameters. The physico-chemical characteristics of sunflower oil were evaluated by drawing out the oil samples (75 ml) from the fryer at an interval of 2 h. The parameters evaluated were refractive index, colour, total polar material, free fatty acids, iodine value and peroxide value. There was a gradual increase in refractive index and colour with time of frying which was found to be significant ($P < 0.05$) in the latter. Iodine value decreased significantly ($P < 0.05$) with time of frying. Peroxide value first increased up to 12 h of frying and then decreased. After 10 h of frying, the total polar materials were 26.9%, indicating the need for replenishment or refinement of sunflower oil. Total polar material correlated well ($r > 0.97$) with colour, free fatty acids, peroxide value and time of frying. Free fatty acids correlated well ($r > 0.96$) with colour, total polar material and duration of frying. Fit of the equations was determined for total polar material and free fatty acids as a function of the respective correlated parameters.

Keywords: Sunflower oil; Quality; Frying; Free fatty acids; Total polar material; Iodine value; Peroxide value; Refractive index; Colour

1. Introduction

Deep fat frying is a process wherein a food is allowed to be immersed and held in hot fat for the purpose of cooking. Fried foods have a distinct appeal, attributed to their characteristic colour, flavour, aroma and crispness. The quality of frying medium has a lot of bearing on the quality of fried product.

The repeated use of oil could affect the shelf life of fried foods due to the development of rancidity in the frying oil taken up by the products (Che Man & Jasvir, 2000). The repeated use of oil at high temperatures results in several oxidative, polymerization and thermal degradation reactions leading to changes in their physical, chemical, nutritional and sensory properties (Gloria & Aguilera, 1998). The intensity of these reactions depends on duration, method of heat treatment, frying medium and type of product (Blumenthal, 1991). Some of these changes are important to impart sensory quality characteristics in fried foods (Shahidi, Wanasundara, & Wanasundara, 1997). However, during continuous/repeated use of oil, a stage is reached when functional, sensory and nutritional quality of fat changes to such an extent that obtaining high quality fried products from the frying medium is not possible and it has to be discarded (Stevenson, Vaisey-Genser, & Eskin, 1984). Many of the degradation products of the edible oils are harmful to health as they destroy vitamins, inhibit enzymes and could cause mutations or gastrointestinal irritations (Clark & Serbia, 1991).

Therefore, it is essential to monitor the quality of oil to avoid the use of abused oil due to health consequences of consuming foods fried in degraded/abused oil, to maintain the quality of fried foods and to minimize the production cost associated with early disposal of frying medium (Vijayan, Slaughter, & Paul Singh, 1996).

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doi:10.1016/j.foodchem.2007.06.023

Oxidative alteration in the frying medium is caused due to release of moisture from the food, atmospheric oxygen entering the oil whereas thermal alteration is caused due to higher frying temperatures (Tseng, Moreira, & Sun, 1996). The rate of formation of decomposed products depends on the type of food being fried, design of the fryer and the operating conditions (Stevenson et al., 1984). Frying of meat results in leaching of fat from meat into the frying medium thereby mixing with it (Landers & Rathman, 1981; Weiss, 1970).

During continuous deep fat frying, the quality of fat deteriorates to a level where it is unsuitable for frying operations. Thus, ascertaining the stage at which the fat deteriorates to the extent that it must be discarded, replenished or treated with adsorbents is of vital importance. Frying is a method that is often used for fish cooking. Sunflower oil is a commonly employed frying medium (Blumenthal & Stier, 1991). Therefore, this study was undertaken to assess the quality of sunflower oil during frying of fish (*Catla catla*).

The objective of this study was to evaluate the quality changes in sunflower oil at 180 °C and, to investigate the correlation between the different studied parameters as well as determine the time when oil reaches its maximum safe use during fish frying.

2. Materials and methods

2.1. General Supplies

Refined sunflower oil, 'Sundrop' manufactured by Agro Tech Food Ltd., Secunderabad, India was purchased from the local market. Red chilli powder (MTR brand), turmeric powder (MTR brand) and fish spice mix (MDH brand) were also purchased from the local market. Mini master fryer (M/S Continental India, Bangalore) was used for carrying out frying operations. The capacity of fryer was 6 l, with thermostatic temperature control from 0 to 300 °C. All the chemicals used were of analytical grade.

2.2. Fish frying

Fresh fish (*C. catla*) pieces were procured from the local market, thoroughly washed and cleaned manually. The pieces were marinated for 2 h by dipping in a solution containing 0.5% citric acid and 6% salt. Thereafter, the solution was drained and fish pieces smeared with spices (turmeric @2 g/kg, chilli powder @7 g/kg, fish spice mix @7 g/kg) and corn starch (25 g/kg), and fried for 6 min in 5 l of sunflower oil maintained at 180 °C. A total of 8 kg fish was fried in 14 h.

2.3. Oil samples

After every 2 h of frying, 75 ml oil was withdrawn for analysis until 14 h of frying. The volume of oil was not replenished during frying operations.

2.4. Oil analysis

The oil quality parameters viz, refractive index, colour, free fatty acids, total polar material, peroxide value, iodine value were determined for fresh sunflower oil as well as after every 2 h until 14 h of frying.

An Abbe Refractometer Model R-8 was used for measuring the refractive index, according to AOAC (1984). Few drops of the moisture free sample filtered through filter paper, were placed on the lower prism. The prisms were closed and tightened firmly with the screw head and allowed to stand for 1–2 min. Alidade was moved forward and backward until the borderline dividing the light and dark portion became sharp and colourless and it fell on the point of intersection of crosshairs. For each sample, three readings were taken. Approximate temperature corrections were made by using the formula, $R = R' + K(T' - T)$, where R is the reading reduced to standard temperature, R' is the reading obtained at temperature T', T is the standard temperature and $K = 0.000385$.

Colour was measured using the Lovibond tintometer method as described in Indian Standards (1964). Moisture free sample filtered through filter paper was taken in a cuvette and placed in the provided space in tintometer. The reflected colour of the oil was matched by trying different combinations of red and yellow slides till the reflected colour matched with the slide colour combination. The yellow and red value of the slide colour was taken and repeated thrice for each sample.

Total polar materials were determined using the column chromatography method as described by Billek, Guhr, and Waibel (1978). Silica gel (0.063–0.200 mm) was dried at 160 °C for 4 h. The gel was weighed in a 500 ml flask and water was added to make a 5% (w/w) mixture. Glass column (length 45 cm and diameter 2.1 cm) was taken for chromatography. For the elution of the column, two different solvents were used. Solvent 1 consisted of petroleum ether (40–60 °C) and diethyl ether in the ratio of 87:13, v/v. Pure diethyl ether was used as solvent 2. A wad of cotton was tightly packed over the outlet by running 30 ml of solvent 1 through it. Silica gel was slurried in solvent 1 (80 ml) and introduced into the column. The gel was then covered with a 1 cm layer of acid washed and calcined sea sand and the supernatant was drained off to the level of sand. One gram (±1 mg) of the filtered oil was weighed in 25 ml beaker and dissolved in 10 ml of solvent 1. Then it was introduced onto the packed column and the solution was drained off until its level reached the top of the sand layer and was collected in a 250 ml beaker (fraction 1). The 25 ml beaker was rinsed with 5 ml of solvent 1 and the rinsings were poured into the column. The column was eluted with 150 ml of solvent 1 and rinsings were poured into the column. Thereafter, the column was eluted with 150 ml solvent 1 and the eluate was collected in the same beaker as fraction 1. This was followed by eluting the column with 150 ml of solvent 2. This eluate was collected in second beaker (fraction 2). The two fractions were separately added in portions to pre-weighed 100 ml round bottom flasks and the solvent was distilled off at a temperature of 60 °C using rotary vacuum evaporator. The flasks were weighed and the contents were reported as percentage of the total fat weight. Fraction 1 was the amount of unaltered triglycerides and fraction 2 was the total polar materials.

Indian Standards (1964) method was used for determining free fatty acids. Oil sample weighing 7.05 ± 0.05 g was taken in a 200 ml conical flask. Freshly neutralized 50 ml hot ethyl alcohol and phenolphthalein indicator, 1 ml were added. The mixture was boiled for 5 min and titrated as hot as possible against standard NaOH solution. Formula $28.2 \times (VN/W)$ calculated free fatty acids in terms of oleic acid percent by weight where V is the volume in ml of NaOH solution, N is the normality of NaOH solution and W is the weight of the oil sample in g.

The iodine value was determined using Wij's method, by following the procedure given in Indian Standards (1964). In a 500 ml iodine flask, sample was weighed accurately between 0.1700 and 0.2125 g, 25 ml of carbon tetrachloride was added to it and the mixture was agitated to dissolve the oil. To the solution, 25 ml of the Wij's solution was added and glass stopper was replaced after wetting with potassium iodide solution; swirled and allowed to stand for 30 min. A blank test was carried out simultaneously under similar experimental conditions. After standing, 15 ml of potassium iodide solution and 100 ml of water were added and the liberated iodine was titrated against standard sodium thiosulphate solution while swirling the contents continuously to avoid any local excess until the colour of the solution was straw yellow. Thereafter, one ml of starch solution was added and the titration continued until the blue colour disappeared after thorough shaking with stopper on. Iodine value was calculated using the formula $12.69(B - S)N/W$. Where B is the volume in ml of standard sodium thiosulphate solution required for the blank; S is the volume in ml of standard sodium thiosulphate required for the sample, N is the normality of the standard sodium thiosulphate solution and W is the weight of the material taken for test in g.

AOAC method (1984) was employed for the determination of peroxide value wherein, 5.00 ± 0.05 g of sample was taken in a glass-stoppered erlenmeyer flask and then 30 ml of acetic acid–chloroform (3:2) solution was added. The flask was swirled until the sample dissolved in the solution. Saturated potassium iodide solution (0.5 ml) was added and allowed to stand with occasional shaking for 1 min and then 30 ml of distilled water was added. It was titrated against standard sodium thiosulphate solution until the yellow colour almost disappeared. Then about 0.5 ml of starch indicator solution was added. Titration was continued until the blue colour just disappeared. Blank was also determined under similar conditions. Peroxide value (milli equivalents/kg sample) was calculated using the formula $S \times N \times 1000$/g sample, where $S =$ ml $Na_2S_2O_3$ (blank corrected) and $N =$ normality of $Na_2S_2O_3$ solution.

2.5. Data analysis

Statistical analysis of the data for comparison, correlation and regression was carried out using MS-Excel, Minitab version 1.4, Costat of CoHort and Curve expert 1.3.

3. Results and discussion

3.1. Evaluation of physical characteristics of fried oil

The physical characteristics, refractive index and colour of the refined sunflower oil during deep fat frying of fish, have been given in Table 1.

The initial value for refractive index of sunflower oil was found to be 1.4678 (Table 1). Even after subjecting the oil to 14 h of frying, there was no significant ($P < 0.05$) increase in the refractive index values i.e. from 1.4678 to 1.469. These findings were supported by the data presented on the study pertaining to the dynamics of a canola based shortening degradation (Paul & Mittal, 1996). The work carried out on vegetable oils by Vidyasagar, Arya, Premavalli, Parihar, and Nath (1974) and Sulthana and Sen (1979) indicated slight increase in Butyro refractometer readings due to heating. Since, in this study there was only a slight increase in refractive index and after statistical analysis it was found to be non-significant ($P > 0.05$), so refractive index cannot be used as a marker for judging oil quality during fish frying.

The change in colour was visible to the naked eye and this was supported by the tintometer values obtained as shown in Table 1. The initial tintometer reading for the refined sunflower oil indicated yellow value of 0.6 and red value of 0.0. After every 2 h of frying the change in yellow value was significant ($P < 0.05$) and finally after 14 h of frying the value was found to be 30.85. The red value of colour presented in Table 1 indicated that the values were not significantly different ($P > 0.05$) during 0–2 and 4–8 h of frying. Earlier workers (Tan, Ong, Berger, Oon, & Poh, 1985) have also reported the increase in intensity of colour change of the oil from yellow to an orange brown during frying. After evaluating the yellow and red values

Table 1
Physical characteristics of refined sunflower oil during deep fat frying of fish ($n = 3$)

Time of frying (h)	Refractive index	Colour yellow	Colour red
0	1.4678	0.6^{h}	0^{e}
2	1.4679	1.3^{g}	0^{e}
4	1.4689	3.6^{f}	0.2^{d}
6	1.4689	4.7^{e}	0.2^{d}
8	1.469	8.4^{d}	0.3^{d}
10	1.469	14^{c}	0.75^{c}
12	1.469	18.25^{b}	1.3^{b}
14	1.469	30.85^{a}	1.75^{a}
LSD (0.05)	Non-significant	0.6046393	0.1412133

Mean values with different superscripts within a column differ significantly ($P < 0.05$).

of colour, it was found that there was a significant ($P < 0.05$) increase from 0.6 to 30.85 and 0 to 1.75, respectively after 14 h of frying. This darkening of oil could be observed during frying process even by the naked eye. There are several reasons reported in the literature for the darkening of oil colour upon heating. The colour pigments during frying undergo oxidation and diffuse from the food into the oil (Fritsch, 1981; Yoon, Kim, Kim, & Kwan, 1987). The coloured degradation products formed by the interaction of food components with oil may contribute to the darkening of the oil (Takekoka, Gerhard, & Das, 1997). The other reasons that could lead to colour change may the presence of suspended charred particles (Paul & Mittal, 1997), absorption of colour from the fried food (Che Man, Suhardiyano, Asbi, Azudin, & Wei, 1996) and polymerization reactions at high temperatures (Nasirullah, 2001).

3.2. Evaluation of chemical characteristics of fried oil

The chemical parameters, total polar materials, free fatty acids, iodine value and peroxide value have been determined at different intervals of frying and represented in Table 2. Total polar material is a chemical parameter that reflects the high temperature degradation of frying oil (Warner & Gupta, 2003). The value of total polar materials in sunflower oil increased significantly ($P < 0.05$) at an interval of 2 h during frying and reached 34.2% from an initial value of 3.58% after 14 h. Earlier workers (Gere, 1982; Premavalli, Madhura, & Arya, 1998) have also reported an increase in total polar material with advanced heating time which is in concurrence with our findings. The total polar material reached a value of 26.9% after 10 h of frying. As per the standards, oil with total polar materials more than 25% is not fit for consumption (Paul & Mittal, 1996). Therefore, total polar materials could be taken as a marker for assessing oil quality.

Free fatty acid value is taken as a rancidity parameter to assess the quality of vegetable oils. An initial value of 0.05% as oleic acid was observed in the case of refined sunflower oil, which has been used as the frying medium in the present study. From the values presented in Table 2 it could be observed that significant ($P < 0.005$) change was at 2, 8, 10, 12 and 14 h of frying and the final value after 14 h of frying was 0.585% (oleic acid). There have been reports regarding on increase in free fatty acids value due to heating of groundnut (Sulthana & Sen, 1979) and mustard (Premavalli et al., 1998) oils. Oxidative and thermal degradation taking place in unsaturated fatty acids is responsible for the production of free fatty acids (Nasirullah, 2001). In addition, an increase in free fatty acids has been known to result from hydrolysis of trigycerides, triggered by infusion of moisture from the food into the oil and its oxidation (Fritsch, 1981). However, determination of free fatty acids by titration is a quantitative method so it is difficult to make out the extent to which the increase in free fatty acids is due to oxidation or hydrolysis (Fritsch, 1981).

Table 2
Chemical characteristics of refined sunflower oil during deep fat frying of fish ($n = 3$)

Time of frying (h)	Free fatty acids (% oleic acid)	Total polar material (%)	Peroxide value (meq/kg)	Iodine value
0	0.05^{f}	3.58^{h}	0.1^{h}	126.44^{a}
2	0.108^{e}	6.99^{g}	1.64^{g}	125.78^{a}
4	0.11^{e}	11.65^{f}	13.51^{f}	124.27^{b}
6	0.122^{e}	15.97^{e}	17.36^{e}	121.63^{c}
8	0.3095^{d}	22.68^{d}	21.27^{d}	121.19^{d}
10	0.359^{c}	26.9^{c}	26.63^{b}	120.40^{e}
12	0.4925^{b}	29.98^{b}	28.98^{a}	118.93^{f}
14	0.585^{a}	34.2^{a}	24.88^{c}	117.42^{g}
LSD (0.05)	0.0395985	0.5153803	1.0301316	0.6688405

Mean values with different superscripts within a column differ significantly ($P < 0.05$).

The iodine value as a quality parameter index during frying of fish in refined sunflower oil was evaluated at 2 h intervals during frying up to 14 h and the values have been presented in Table 2. The initial iodine value of fresh oil was 126.44 which decreased significantly ($P < 0.05$) to 117.42 after 14 h of frying. Statistical analysis of the data revealed a significant ($P < 0.05$) variation at 4, 6, 8, 10, 12 and 14 h of frying. The decrease in iodine value with time of frying could be attributed to the changes in fatty acids taking place with duration of frying (Tynek, Hazuka, Pawlowicz, & Dudek, 2001). Reblova, Kudrnova, Trojakova, and Pokorny (1999) also reported a decreasing trend in iodine value of the oil during deep fat frying.

In order to assess the oxidative changes in refined sunflower oil during fish frying peroxide value was determined and has been presented in Table 2. Significant ($P < 0.05$) differences were observed in peroxide value at different time intervals. Peroxide value increased from 0.1 meq/kg in fresh oil to 28.98 meq/kg in 12 h fried oil. After 14 h the value decreased to 24.88 meq/kg. The decrease in peroxide value after 14 h of frying may be attributed to the instability of peroxides (Fritsch, 1981). Other workers also (Susheelamma, Asha, Ravi, & Vasanth Kumar, 2002; Sulthana & Sen, 1979) have reported increase in peroxide value of oil upon heating.

3.3. Relationship among quality parameters

The relationships among quality parameters were evaluated by using MS-Excel, Minitab version 1.4, Costat, Curve Expert 1.3 software. A correlation of total polar material was observed with duration of frying ($r = 0.998$, $SE = 0.7555$), FFA ($r = 0.982$, $SE = 2.244$), colour Y ($r = 0.993$, $SE = 1.414$) and peroxide value ($r = 0.971$, $SE = 3.133$). Apart from the total polar material, free fatty acids correlated well with the duration of frying ($r = 0.968$, $SE = 0.066$) and colour Y ($r = 0.99$, $SE = 0.032$). Regression analysis was carried out and the fit of equations were determined for the data with maximum correlation coefficient and minimum standard error.

Total polar material exhibited polynomial fit (Fig. 1), exponential fit (Fig. 2), logarithmic fit (Fig. 3) and reciprocal quadratic fit (Fig. 4) with duration of frying, yellow value of colour, free fatty acids and peroxide value, respectively. Polynomial fit was observed between free fatty acids and duration of frying (Fig. 5) while a quadratic fit (Fig. 6) was observed between free fatty acids and yellow colour values.

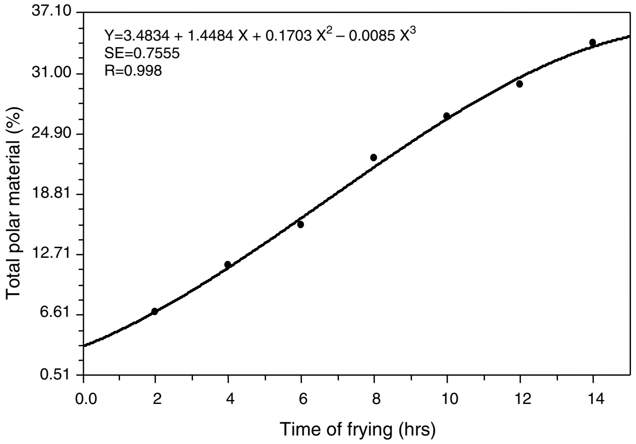

Fig. 1. Polynomial fit (third degree) for total polar material and time of frying.

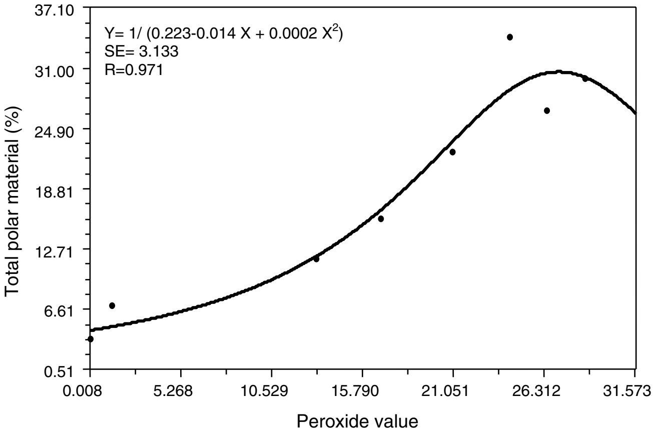

Fig. 4. Reciprocal quadratic fit for total polar material and peroxide value.

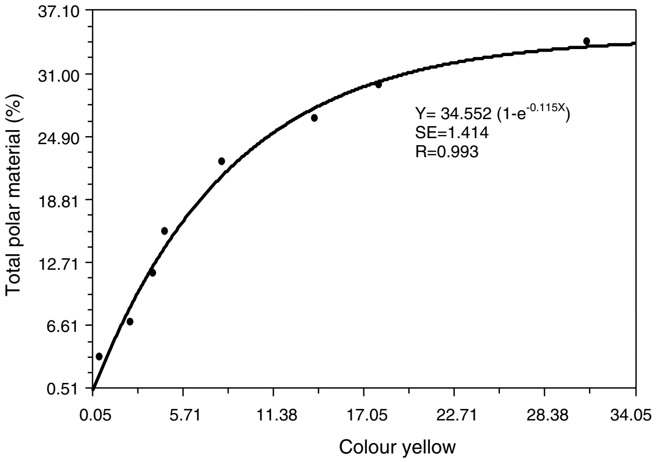

Fig. 2. Exponential fit for total polar material and colour yellow.

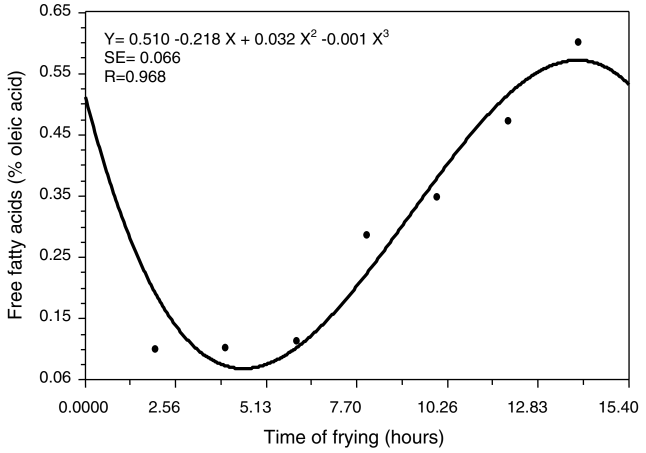

Fig. 5. Polynomial fit (third degree) for free fatty acids and time of frying.

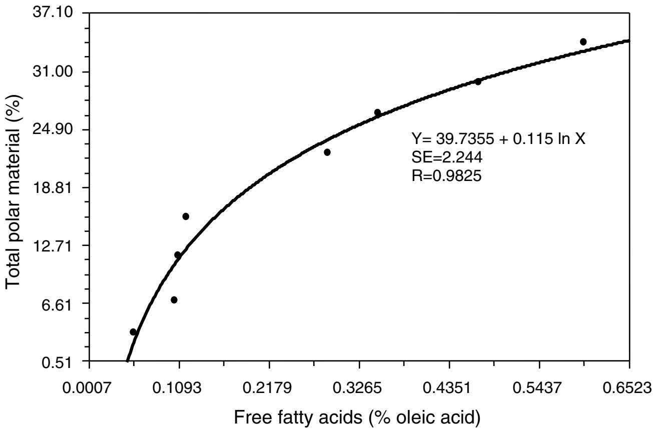

Fig. 3. Logarithmic fit for total polar material and free fatty acids.

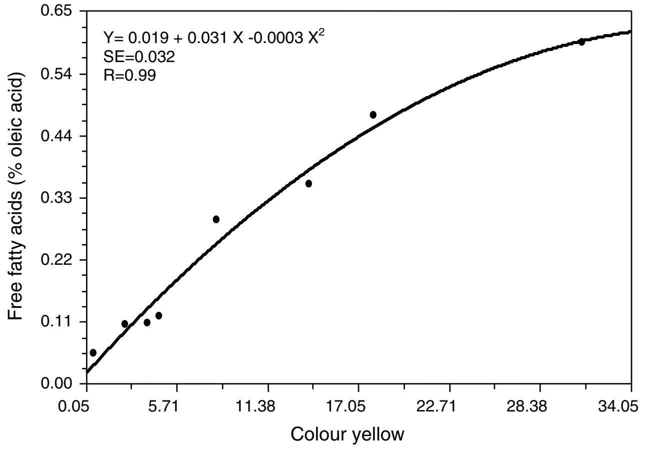

Fig. 6. Quadratic fit for free fatty acids and colour yellow.

Total polar material could be determined using the following equation:

$$\mathrm{TPM} = 2.13 - 2.64\ \text{colour R} - 0.096\ \text{colour Y} + 2.19\ \text{time(h)} + 14.7\ \text{free fatty acids},$$

where $\mathrm{SE} = 0.9642$ and $r^2 = 0.997$.

Similarly,

$$\text{Free fatty acids} = -0.036 + 0.1019\ \text{colour R} + 0.0263\ \text{total polar material} - 0.048\ \text{time(h)} + 0.00798\ \text{colour Y},$$

where $\mathrm{SE} = 0.04076$ and $r^2 = 0.982$.

4. Conclusion

Analysis of quality parameters, physical as well as chemical during fish (*C. catla*) frying, revealed that the quality of frying oil started deteriorating with time as found by studying oil quality parameters viz., refractive index, colour, total polar materials, free fatty acids, iodine value and peroxide value. The total polar material could be taken as an important marker to assess the quality characteristics of frying medium. From the study it was revealed that the cut-off value of total polar material for assessing the quality index (total polar material > 25%) was obtained after 10 h of frying. Statistical analysis revealed a significant ($P < 0.05$) correlation between the total polar materials and other quality index parameters like free fatty acids, colour and peroxide value. Fit of equations have been designed for the calculation of total polar materials and free fatty acids that are important parameters in deciding the safe limit of frying oil with respect to time of frying and colour. The results suggested that as the value of total polar material exceeded 25% after 10 h of fish frying the oil should be discarded, replenished or treated with suitable adsorbents after the same.

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