Sepiolite: An Effective Bleaching Adsorbent for the Physical Refining of Degummed Rapeseed Oil

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ABSTRACT: The efficiency of Turkish sepiolite in bleaching degummed rapeseed oil has been investigated. Experimental results indicate that the bleaching efficiency is more dependent on the ratio of sepiolite to oil than on operating parameters such as contact time and temperature. An increase in the sepiolite dosage reduces the color bodies of the rapeseed oil. Its effect on oxidation state, however, is complex and related to both primary and secondary oxidation products. The removal of impurities such as chlorophyll a, β-carotene, and phosphorus increases with increasing sepiolite dosage and reaches a maximum at 1.5% sepiolite addition and 100°C bleaching temperature. Chlorophyll a, β-carotene, and phosphorus adsorptions can be described by a mechanism involving surface area and porosity of acid-activated sepiolite as key variables. The sorption is also independent of the polarity of the adsorbate molecules. Direct comparison of activated sepiolite with the commercial bleaching earth Tonsil 210 FF shows that in some respects sepiolite offers significant advantages.

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KEY WORDS: Bleaching adsorbent, physical refining, rapeseed oil, sepiolite.

In the process of refining edible oils, a clay adsorbent typically is mixed with the oil under specified conditions to remove undesirable color bodies (e.g., carotene, chlorophyll, and their decomposition products) and other contaminants (trace metals, peroxides, phosphatides). Removal of these impurities requires the use of strongly adsorbing materials. Bentonites (layered clay minerals) in their natural state are known to have a limited ability to bleach coloring matter during industrial preparation of vegetable fats and oils. The commercial clays are usually acid-activated to improve their bleaching property, which results in the delamination of their structure (1–3).

Sepiolite is a natural fibrous clay mineral with a hydrated magnesium silicate formula $[(Si_{12})(Mg_8)O_{30}(OH)_6(OH_2)_4 \cdot 8H_2O]$ (4). It has a high decolorization capacity because of its high specific surface area, porosity, and surface activity, and its ability to form high and stable viscosities at low solids concentrations. The sorption ability of sepiolite is mainly ascribed to its high surface area (5). Thus, it is commonly used in oil refining, wastewater treatment, in the removal of odor, as a drug and pesticide carrier, and in the manufacture of paper and detergent (6–10).

Similar to other chain-type silicate minerals, sepiolite contains a continuous 2-D tetrahedral sheet but without continuous octahedral sheets. This unique microfibrous structure, with interior channels of 3.6 × 10.6 Å, permits adsorption of organic and inorganic ions (11). The presence of micropores and channels (Fig. 1) along with the elongated nature of the particles and the fine particle size accounts for the high surface area and adsorptivity. Under natural conditions, the channels are filled with zeolitic water that is loosely hydrogen-bonded to the basal oxygen planes. The zeolitic water in the channels does not show acidic properties, unlike water coordinated with exchangeable cations in charged layer silicates. The structural water coordinated to the edge of the octahedral ribbons also is

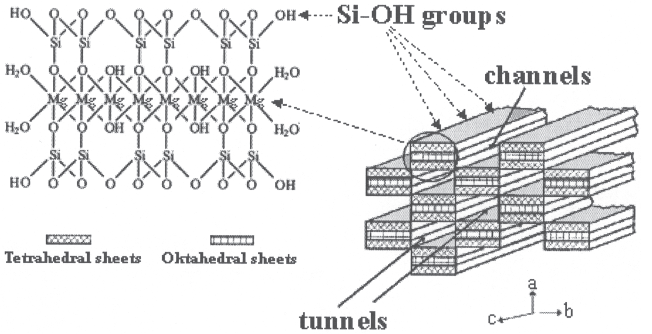

FIG. 1. Schematic illustration of sepiolite structure.

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*To whom correspondence should be addressed. E-mail: esabah@aku.edu.tr not acidic, and natural sepiolite presents a basic surface with a limited catalytic activity. Sepiolite has a total specific surface area of 800–900 m^2/g, of which theoretically 400 m^2/g is external and 500 m^2/g is internal (6,12).

The purpose of the present study was to undertake preliminary laboratory investigations on the effectiveness of acid-activated sepiolite from Turkey as a bleaching adsorbent in physical refining of rapeseed oil and also to determine the optimal dosage of sepiolite for commercial applications. The performance of sepiolite in removing carotene, chlorophyll, phosphorus, acidity, and oxidation products was compared with a standard activated clay, Tonsil 210 FF.

EXPERIMENTAL PROCEDURES

Materials. Pressed and filtered rapeseed oil was obtained from Bio-Ölwerk Magdeburg GmbH (Magdeburg, Germany). To eliminate the influence of different crude oil qualities on the bleaching process, experiments were carried out with rapeseed oil that had been degummed with water and acid, followed by drying. The composition of the degummed rapeseed oil was as follows: FFA (mg KOH/g), 1.3 ($n = 3$); PV (mequiv O_2/kg), not detected; anisidine value, 2.8; chlorophyll a (ppm), 4.85; β-carotene (ppm), 42.3; phosphorus (ppm), 29.7; color: Lovibond Red 5 1/4″, 3.8; Lovibond Yellow 1″ = 70.

The sepiolite sample from Türktaciri-Sivrihisar, located in midwest Turkey, that was used in this study was received from Anadolu Industrial Minerals Co. This beige sepiolite (BS) with a size of approximately minus 2 mm was ground to minus 75 µm in a vibratory mill to produce an average particle size (d_{50}) of 14.41 µm as determined by HELOS-Zetasizer; the sample was activated with 1 M nitric acid, which was purchased from Roth (Karlsruhe, Germany) and specified to be of high purity. The specific surface area of activated BS was measured by N_2 adsorption isotherm at 77.4 K in a Micromeritics-Flowsorb II-2300 volumetric adsorption system. The samples were degassed at 60°C for 30 min before measurement.

The Tonsil 210 FF used as standard activated clay was purchased from Süd-Chemie A.G. (Munich, Germany). Table 1 lists the relevant analytical data for the acid-activated sepiolite and Tonsil 210 FF.

In all experiments distilled water was used.

Methods. The following procedure was used to activate natural sepiolite: 300 g of the ground BS was placed in a 5-L, 3-necked round-bottomed flask; 3000 mL of 1 N HNO_3 solution was added so that the acid/sepiolite ratio (wt/vol) is 10%. The stirrer and reflux condenser were attached to the flask and maintained at room temperature for 1 h with stirring at 350 revolutions per min. After activation, the sepiolite suspension was transferred to a 5-L beaker and rinsed twice with distilled water until pH 5.8 was achieved. The liquid fraction was filtered under vacuum; the acid-activated BS was dried in an oven at 60°C for 15 h and reground using a centrifugal ball mill (RETSCH S 100) to pass through a 75 µm sieve for the bleaching tests.

The percent acidity measurement of acid-activated sepiolite [the N.G.D. C-10-1976, as given in Rossi *et al.* (13)] is based on titration with a 0.1 N NaOH solution using phenolphthalein as indicator. The apparent bulk density (ABD) of the sepiolite sample was determined according to the German version of European Standard EN 1097-3 (14). A sepiolite sample of minus 14.75 µm was dried in an oven at 110 ± 5°C to a constant mass. An empty, dry, clean 1-L measuring cylinder was weighed (m_1) and filled loosely with sepiolite up to the top. The filled cylinder was then weighed (m_2). The ABD for each sepiolite sample was calculated as ABD (g/L) = $(m_2 - m_1)/V$, where V is the volume of cylinder.

TABLE 1
Physical and Chemical Characteristics of Bleaching Clays

Parameters	Acid-activated sepiolite	Tonsil 210 FF[a]
Apparent bulk density	223 g/L	510 g/L
Free moisture (2 h, 110°C)	~10 %	~10%
Loss on ignition (predried, 2 h, 1000°C)	~10%	6–8%
pH (10% suspension)	5.8	3.2
Acidity	0.11%	1.0 mg KOH/g
Surface area (BET)	280 m^2/g	210 m^2/g

[a] These values are supplied by the manufacturer, Süd Chemie (Munich, Germany).

The vacuum bleaching tests were carried out in a 1-L, 4-necked round-bottomed flask that was heated from the outside. The oil was agitated with a stirrer at approximately 400 rpm throughout the bleaching process. The temperature was controlled with a mercury thermometer. A vacuum pump was used to maintain the necessary pressure at 40 mbar. After bleaching and cooling to 70–80°C, the oil was passed through a pressure filter. All bleaching experiments were carried out at temperatures of 80 and 100°C whereas the standard bleaching tests were performed at 90°C.

Analyses. FFA content in the unbleached and bleached oil was determined according to the German Standard Methods for the Analysis of Fats and Other Lipids of the German Society for Fat Research (DGF; Deutsche Gesellschaft für Fettwissenschaft) DGF C-V 2(81) (15). PV, which represents a quality assessment for identifying the extent of primary oxidative deterioration in fats and oils, was measured by AOCS Official Method Cd 8b-90 (16). Values for both FA and PV in this text are based on the average of three measurements.

The anisidine value (AnV) is defined by convention as 100 times the O.D. of a solution following the reaction of 1 g of oil or fat in 100 mL of a mixture of solvent and reagent measured in a 1-cm cell as described in the German Standard Method DGF C-VI 6e(84) (17). The amount of chlorophyll a remaining in the oil was determined by AOCS method Cc 13d-55 (16). The β-carotene content of the oil was measured at 450 nm according to the ÖHMI L 004-13 Method (18), and the color of the oil was determined on a Lovibond Tintometer (Model PFX990) according to the AOCS Official Method Cc 13d-55 (16). The phosphorus content of the oil was determined by German Standard Method DGF C-VI 4(61) (19).

The oxidation value of an edible oil, which represents its quality, depends directly on the quantity of oxygen absorbed.

TABLE 2
Analysis of Rapeseed Oil After Bleaching at 80°C for Different Contact Times Using Various Dosages Sepiolite

	0.5% Sepiolite			1.0% Sepiolite			1.5% Sepiolite			1.0% Tonsil[a]
Parameters	15 min	20 min	25 min	15 min	20 min	25 min	15 min	20 min	25 min	20 min
					Bleach contact time					
FFA (mg KOH/g)[b]	0.84	0.84	0.81	0.75	0.80	0.74	0.90	0.72	0.79	1.04
PV (mequiv O_2/kg)[b]	9.67	9.92	9.74	7.82	9.10	8.80	10.02	7.85	7.27	2.71
Anisidine value	3.6	4.1	2.9	3.9	5.5	3.0	5.3	2.9	4.4	6.6
Chlorophyll a content (ppm)	3.08	4.25	3.66	0.52	2.37	2.79	1.09	0.25	1.88	0.19
β-Carotene content (ppm)	13.9	13.7	12.6	4.53	4.94	4.67	3.17	1.95	2.55	9.40
Phosphorus content (ppm)	21.2	17.2	17.6	7.98	5.66	5.10	1.04	2.17	3.13	15.60
Oxidation value	22.94	23.90	22.38	19.54	23.70	20.06	25.34	18.06	18.94	12.02
Color										
Lovibond Red 5 1/4″	3.2	3.2	3.2	1.6	1.5	1.4	0.6	0.9	0.8	1.0
Lovibond Yellow 1″	70	70	70	70	70	70	32	13	19	38

[a] Standard bleaching test with Tonsil 210 FF at 90°C.
[b] Average of three measurements.

The total oxidation value (Totox) is being increasingly used as a characteristic value for the overall degree of oxidation of a TG. Totox is calculated from the PV and AnV as follows: Totox = 2 PV + AnV (20). Totox values of >30 are indicative of oxidized oils and fats.

RESULTS AND DISCUSSION

Bleaching effect of acid-activated sepiolite. The most important properties of activated bleaching clays with respect to adsorptive bleaching of vegetable oils include total acidity, pH, surface area and porosity, degree of acid activation, particle size, and moisture content. Bleaching power appears to be more a function of a clay's bound acidity; therefore, clays having high total acidity and a reasonable level of free acidity are preferred (21,22).

Results of the analysis of the degummed rapeseed oil following treatment with acid-activated sepiolite at different dosages and bleach contact times at 80 and 100°C are shown in Tables 2 and 3; for comparison a commercial industrial bleaching earth (Tonsil 210 FF) was evaluated. The range of temperature and amount of sepiolite used in bleaching of oil do not lead to significant changes in the FFA content of the refined rapeseed oil. Ordinarily, bleaching has little effect on the acidity of oils. Neutral or slightly acid clays often reduce the acidity very slightly through preferential adsorption of the FFA (23). Although a low pH with substantial acidity in the clay is desirable for bleaching chlorophyll-containing oil, low pH also has a detrimental effect. There is an inverse ratio between the pH of the clay and FFA increase in the oil during bleaching (24). The reduction observed in the FFA contents at both bleaching temperatures considered in this paper is attributed to the low acidity and higher pH of sepiolite (Table 1). Taking into consideration the bleaching temperature, its associated period, and the weight percentage of sepiolite used in bleaching, the FFA value before bleaching was 1.3 mg KOH/g whereas after treatment with 1% sepiolite for 20 min this value fell to 0.74 mg KOH/g. Interestingly, Tonsil 210 FF reduced this value to 1.04 mg KOH/g. Therefore, moderate or slight surface acidity of sepiolite can be exclusively ascribed to the presence of

TABLE 3
Analysis of Rapeseed Oil After Bleaching at 100°C for Different Contact Times Using Various Dosages Sepiolite

	0.5% Sepiolite			1.0% Sepiolite			1.5% Sepiolite			1.0% Tonsil[a]
Parameters	15 min	20 min	25 min	15 min	20 min	25 min	15 min	20 min	25 min	20 min
					Bleach contact time					
FFA (mg KOH/g)[b]	0.78	0.83	0.83	0.71	0.74	0.73	0.66	0.71	0.68	1.04
PV (mequiv O_2/kg)[b]	10.56	11.70	12.17	8.75	7.62	8.24	5.85	6.60	6.06	2.71
Anisidine value	6.9	4.7	6.3	7.1	5.0	5.2	5.8	6.4	6.3	6.6
Chlorophyll a content (ppm)	3.32	4.32	4.40	1.43	0.69	0.71	0.16	1.75	0.53	0.19
β-Carotene content (ppm)	11.2	11.6	10.7	3.82	4.71	2.18	1.60	3.17	1.92	9.40
Phosphorus content (ppm)	17.9	14.8	12.1	7.93	4.44	6.11	1.99	1.11	1.95	15.60
Oxidation value	28.02	28.10	30.64	24.60	20.24	21.68	17.50	19.60	18.52	12.02
Color										
Lovibond Red 5 1/4″	3.6	3.2	2.8	1.6	1.4	1.5	0.8	1.1	1.0	1.0
Lovibond Yellow 1″	70	70	70	38	36	32	12	16	10	38

[a] Standard bleaching test with Tonsil 210 FF at 90°C.
[b] Average of three measurements.

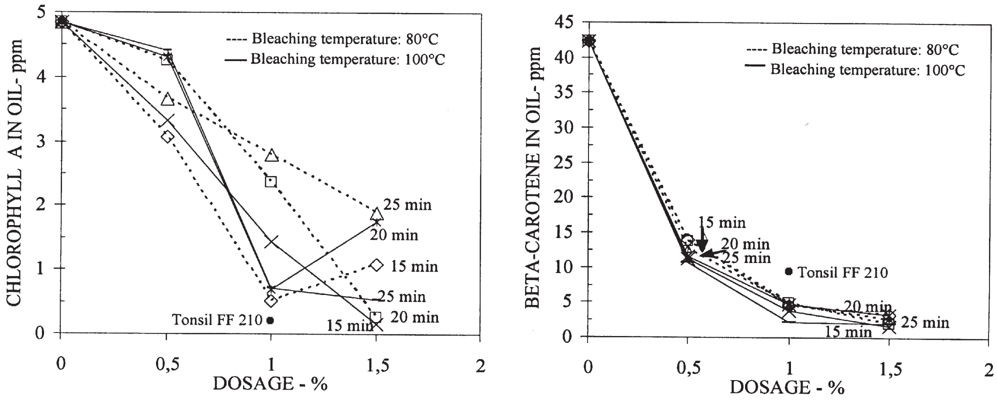

FIG. 2. Effect of acid-activated sepiolite dosage on adsorption of chlorophyll a and β-carotene at different contact times and temperatures.

Lewis sites. The Lewis acid sites of sepiolite may exhibit more OH^- groups than high-acidity sepiolite does for neutralizing FA and also presumably a major cause of reduction observed in the FFA possibly through their preferential adsorption during bleaching operation. Again, whereas clays with high activity exhibit a slight increase in the FFA value of the oil (25), no change was observed on bleaching with sepiolite even at higher dosages.

The production of oil with acceptable oxidative stability requires careful control of refining process conditions. The peroxides and hydroperoxides are responsible for primary oxidation. Clays can both adsorb peroxides and catalyze peroxide through degradation into secondary oxidation products (13). Tables 2 and 3 show the variation of PV and AnV in rapeseed oil with sepiolite dosage at three different contact times and bleaching temperatures of 80 and 100°C.

As shown in Tables 2 and 3, the PV of the oil bleached with activated sepiolite is governed by bleaching temperature and amount of sepiolite rather than bleaching time. The zero PV in the degummed oil increases to a maximal value of 12.17 mequiv O_2/kg after treating it with 0.5% sepiolite and drops to 5.85 mequiv O_2/kg at a maximum sepiolite dosage of 1.5% and 100°C bleaching temperature and 15 min of bleaching time. On the other hand, the standard test with 1% Tonsil 210 FF addition yields a PV of 2.70 mequiv O_2/kg. Data in Tables 2 and 3 show that reaction of rapeseed oil with 0.5% sepiolite does not lead to any changes in oil color; this indicates that 0.5% sepiolite is not capable of inducing any bleaching power. However, the particle size and ABD of sepiolite are presumed to have a major impact on increasing the PV of oil from zero to 9.0 mequiv O_2/kg at 1.0 and 1.5% sepiolite additions. The ABD value of clay depends on the amount of void space in the material. Since sepiolite has a finer particle size and lower ABD than Tonsil, the oxygen held in void spaces is released into the oil and increases its PV. However, the observed increase in the PV of oil is reduced by increasing the sepiolite dosage and reflected in the oxidation values presented in Tables 2 and 3. In other words, when the degummed rapeseed oil is treated with larger sepiolite dosages it is very likely that the peroxides and hydroperoxides responsible for primary oxidation are decomposed and transformed into carbonyl compounds and/or polymeric products. This effect alone would reduce the PV of the oil. However, the net effect will be to increase the AnV of the oil, as it is observed in Tables 2 and 3. Comparison of the results shown in Figure 2B reveals that AnV generally are higher for sepiolite-treated oils than for degummed oil and increase with increasing temperature. This situation confirms the fact that activated sepiolite has an accelerating effect on the conversion of primary oxidation products to secondary ones.

The pigment adsorption efficiencies determined by contacting acid-activated sepiolite and Tonsil 210 FF with degummed rapeseed oil are given in Figure 2. Rapeseed oil contains large amounts of pigments and thus is more difficult to bleach than other vegetable oils (26). Despite this fact, the ability of sepiolite to adsorb color pigments is rather high. But the success achieved by sepiolite in removing β-carotene compared with

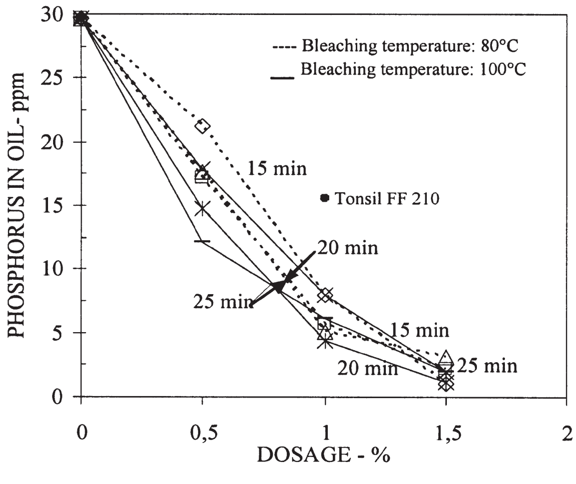

FIG. 3. Effect of acid-activated sepiolite dosage on adsorption of phosphorus at different contact times and temperatures.

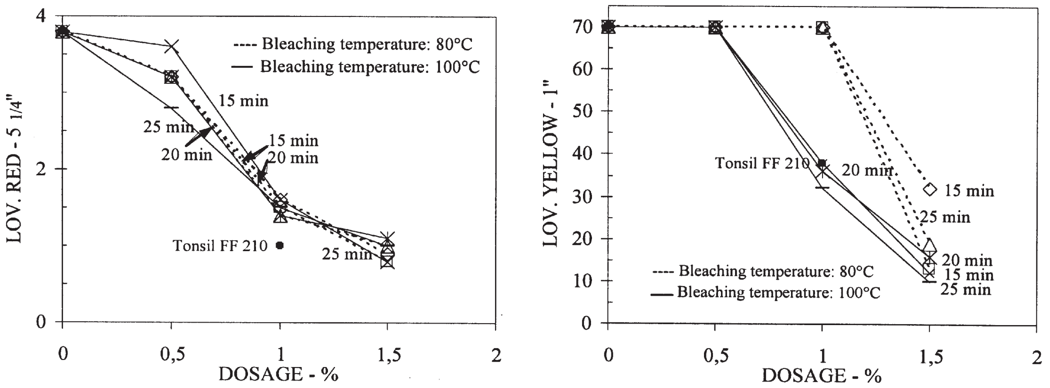

FIG. 4. Decolorization effects of acid-activated sepiolite dosage at different contact times and temperatures.

Tonsil 210 FF was also approached in the case of chlorophyll a by using 50% more sepiolite than Tonsil 210 FF. The low adsorption of chlorophyll at low sepiolite dosages is due to autooxidation process during bleaching being much more sensitive than carotenes in the order of contact temperature, timeABD, and particle size, respectively. Interestingly, for a 1% sepiolite dosage, 100°C bleaching temperature, and bleaching time of 15 min, the adsorption of chlorophyll a is higher than Tonsil 210 FF.

As is evident from Figure 2B, the bleaching temperature and time have no significant effect on the removal of β-carotene from degummed rapeseed oil; however, increasing sepiolite dosage induces substantial removal of β-carotene from the oil. For instance, at 1.0% sepiolite dosage, 100°C bleaching temperature, and 25 min of bleaching time the fractional degree of β-carotene removal is 87%, whereas Tonsil 210 FF under the same conditions removes 78%. Increasing the sepiolite dosage to 1.5% under the same conditions raise the fractional degree of carotene removal to 96%.

Various physicochemical properties of bleaching earth such as acidity, surface area, and pore volume play dominant roles in the removal of color bodies from oil. One or more of these properties may not always participate in the adsorption process. Some experiments indicate that Frosil, a magnesium silicate, does not possess adequate bleaching performance despite its high surface area (*ca.* 300 m^2/g), whereas Laponite, a synthetic hectorite with a surface area of 350 m^2/g, is an efficient bleaching earth for palm oil (3). Synthetic hectorite (magnesium silicate) possesses the opposite characteristics (low pore volume but high surface acidity) (22). Thus bleaching is a complex mechanism and involves more than simple adsorption of color pigments. Some of the color bodies, such as the carotenoids, tend to fade out in bleaching if oxidized by heating (24).

The above results indicate that bleaching efficiency of activated sepiolite for nonpolar impurities in the oil, such as carotenes and chlorophyll, may be related to surface area and porosity rather than to the acidity of sepiolite. Most of the surface area and pore volume is probably generated from internal cavities of mesopore and micropore sizes, which could facilitate the bleaching process. Figure 2 illustrates that the performance of bleaching power of sepiolite may depend on the increase in the number of pores of effective size that accommodate the desired organic molecules. Reduction in the uptake of chlorophyll a and β-carotene at large sepiolite dosages indicates that these molecules are incorporated in the internal micro- and mesopores rather than external surfaces. Radojevic *et al.* (27) report that the affinity of branched hydrocarbons to Goles sepiolite is higher toward internal rather than external surfaces. The oxidative reactions occurring due to the existence of secondary oxidation processes can be also effective in removing pigments and particularly carotenes; the carotenes structurally consist of highly unsaturated hydrocarbon chains

TABLE 4
Adsorptive Properties of Studied Cases

	Contact/degummed rapeseed oil							
	Phosphorus		Chlorophyll		β-Carotene			
Bleaching earths	Content (ppm)	FDB[a] (%)	Content (ppm)	FDB[a] (%)	Content (ppm)	FDB[a] (%)	Lv[b] Red	Lv[b] Yellow
No treatment	29.7		4.85		42.3		3.8	70
Activated sepiolite[c]	4.44	90	0.69	86	4.71	84	1.4	36
Activated sepiolite[d]	1.99	95	0.16	97	1.60	95	0.8	12
Tonsil FF 210[e]	15.60	63	0.19	96	9.40	68	1.0	38

[a] Fractional degree of bleaching (%) = $(A_{unbleached} - A_{bleached})/A_{unbleached} \times 100$, where A = absorbance.
[b] Lovibond.
[c] 1.0 wt% dosage in 150 g oil; 100°C for 20 min.
[d] 1.5 wt% dosage in 150 g oil; 100°C for 15 min.
[e] Standard bleaching: 1.0 wt% dosage in 150 g oil; 90°C for 20 min.

arranged in various cyclic configurations that will heat-bleach and oxidize readily. Their conjugation imparts an orange to red color. Oxidation or heating removes this color.

Acid-activated sepiolite is effective in removing phosphorus (Fig. 3) as well as coloring matter. This probably occurs through a mechanism similar to that of pigment removal, and the process is dependent on surface area and porosity of the activated sepiolite. Acid leaching of sepiolite increases the pore size and hence exposes more of the active adsorption sites. Increasing the sepiolite dosage also increases the number of sites at which phosphorus can be accommodated; phosphorus removal is independent of bleaching temperature and time. Relevant experiments (6,27) reported in the literature using untreated and acid-activated sepiolites have identified a similar mechanism for adsorption of polar molecules such as water, isopropanol, and isobutanol. Figure 3 demonstrates also that activated sepiolite is superior to Tonsil 210 FF for adsorbing phosphorus under all conditions.

Figure 4 shows the influence of sepiolite dosage on the color of degummed rapeseed oil at different bleaching times and temperatures. The color reduction in the Lovibond Red unit is dependent on neither contact time nor temperature at all sepiolite dosages, whereas the color reduction in the Lovibond Yellow unit is dependent on temperature and somewhat on contact time. At 100°C and 1.5% sepiolite dosage, the color of Lovibond Yellow lightened gradually with contact time and decreased to a level less than that achieved with Tonsil 210 FF. However, the latter change is small. Consequently, bleaching conducted at 100°C temperature for 15 min time using 1.5% sepiolite enables the removal of polar and nonpolar impurities and in turn produces lighter color of rapeseed oil than Tonsil 210 FF. Reasonable results were obtained even when using quantities of sepiolite equivalent to that of Tonsil, i.e., 1.0%, particularly in the removal of phosphorus and β-carotene. Table 4 summarizes our results.

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