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COMPARISON OF THE EFFICIENCY OF ADSORBENTS FROM DIFFERENT MANUFACTURERS IN THE PROCESS OF ADSORBATIVE BLEACHING OF COTTON OIL

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Abstract

In this work, the efficiency of adsorbents imported from Switzerland and China in the adsorption bleaching process of refined cottonseed oils obtained by pressing and extraction methods was comparatively studied. The bleaching process was carried out under vacuum at a temperature of 90 °C for 30 minutes, and the adsorbent consumption was varied in the range of 1.0–3.0%. The results showed that with an increase in the adsorbent consumption, the color indicators significantly decreased, but at a rate higher than 2.0–2.5%, the increase in efficiency slowed down, which is explained by the saturation of the adsorption centers. The Swiss adsorbent showed high selectivity in color reduction, reduction of unsaponifiables and peroxide value, and showed a lower oil capacity.

Introduction: In the refining of vegetable oils, the adsorption bleaching stage plays an important role in the removal of coloring pigments, oxidation products, and some unsaponifiables. The bleaching efficiency mainly depends on the surface structure of the adsorbent, the degree of porosity, the number of acidic sites, and the active surface area (Rossi, M., Gianazza, M., Alamprese, C., Stanga, F., 2001; Dijkstra, A.J., 2016).

It has been shown that the efficiency of activated clays in bleaching vegetable oils depends on their mineralogical composition and the density of acidic sites. The authors found that the adsorption of pigments occurs mainly by the mechanism of monomolecular layer formation and that the process is characterized by a Langmuir-type isotherm (Rossi, M., Gianazza, M., Alamprese, C., Stanga, F., 2001).

According to the authors, pigments, phospholipid residues, and secondary oxidation products are selectively adsorbed during the bleaching process. An increase in the consumption of bleaching clay reduces the color index, but after a certain level, a decrease in efficiency is observed, which is explained by the saturation phenomenon of active sites (Dijkstra, A.J., 2016).

It has been proven that increasing the amount of bleaching earth reduces unsaponifiables and oxidation products in the oil. However, increased technological losses have been noted due to the high oil capacity of the adsorbent (Sabah, E., Çelik, M.S., 2005). Comparing adsorbents from different manufacturers, it was found that bentonites with high active surface area are more effective in reducing color, but some cheaper analogues have lower selectivity (Lin, L., Cunshan, Z., Vittayapadung, S., Xiangqian, L., Mingdong, Z., 2014). The decrease in peroxide value during bleaching is mainly due to partial adsorption of hydroperoxides, but this stage cannot completely eliminate oxidation (Zschau, W., 2004).

The authors also showed a direct relationship between the structural properties of the adsorbent and the oil capacity. Adsorbents with high porosity retain pigments better, but also absorb more oil (Boki, K., et al., 1992).

In general, scientific sources show that the efficiency of the adsorbent depends not only on its consumption, but also on its mineralogical composition and surface-chemical properties. Increasing the amount of bleaching earth significantly reduces color, but there is an economic and technological trade-off point, which is often in the range of 2–3% (Dijkstra, A.J., 2016; Lin, L., Cunshan, Z., Vittayapadung, S., Xiangqian, L., Mingdong, Z., 2014).

Keywords: *Cottonseed oil, adsorption bleaching, bleaching earth, bentonite, color indicator*

Materials and methods

The color of the oil was measured using a Lovibond apparatus at a constant 35 yellow units in a 1 cm thick cuvette (Standard 5477–2015).

The acid value was determined by titrating the sample with 0.1 N KOH solution (Standard 5476–80).

The mass fraction of moisture and volatile matter was determined by drying the oil in a drying oven at 105 °C until the mass was constant (Standard 11812–2022).

The mass fraction of non-fatty impurities (precipitation by mass) was determined by the height of the layer separated in a solution of acetone and calcium chloride (Standard 5481–2014).

The soap (qualitative analysis) was determined by heating the sample in distilled water with an indicator (Standard 5480–2023).

The mass fraction of unsaponifiable matter was determined by completely saponifying the sample and extracting the substances insoluble in petroleum ether (Standard 5479–2023).

The peroxide value was determined by the release of iodine under the influence of peroxides in the sample, followed by titration with sodium thiosulfate in the presence of starch (Standard 26593–85).

The flash point was determined by heating the sample in a closed crucible and the combustion behavior of the resulting solvent vapors when brought to a flame (Standard 9287–59).

The presence of solvent (gasoline) in the oil (qualitative analysis) was determined by heating the sample and passing air through a special filter (Uzbek State Standard 816:2015).

The oil capacity of the bleaching earth was determined by calculating the percentage increase in the mass of bleaching earth of a given mass after the bleaching process compared to its initial mass (Arutyunyan, N. S., 1991).

The yield of bleached oil was determined by calculating the mass of the oil after the bleaching process compared to the initial mass (Arutyunyan, N. S., 1991).

The aim of the work is to compare the efficiency of adsorbents from different manufacturers in the process of adsorbative bleaching of cotton oil

The objects of research are cottonseed oil, bleaching earth, bentonite, technological systems, stages of its processing, their analysis and evaluation.

Results

First of all, cottonseed oils obtained by pressing and extraction methods were taken for the study. In this case, the oil was refined and dried, but the bleaching process was not carried out. The physicochemical parameters of these oils are given in Table 1.

These types of oils are sent to the next process, i.e. the bleaching process. In this, adsorbents that are widely used today, i.e. bleaching earths imported from Switzerland and China, were used. The bleaching process was carried out under vacuum at a temperature of 90 °C for 30 minutes. In order to determine the optimal adsorbent consumption for the bleaching process, its amount was determined from 1.0 to 3.0% relative to the mass of the oil. The physicochemical parameters of the obtained refined, bleached cottonseed oils are presented in Tables 2 and 3.

Table 1. *Physicochemical parameters of refined, unbleached cottonseed oil obtained by various methods*

No.	Name of indicators	Unit of measurement	Cotton oil value indicators	
			obtained by pressing	obtained by extraction
1.	Color, constant 35 yellow units:	red unit	12.5	20.0
		blue unit	1.3	2.8
2.	Acid number	mg KOH/g	0.23	0.31
3.	Mass fraction of moisture and volatile matter	%	0.07	0.08
4.	Mass fraction of non-fatty impurities (sedimentation by mass)	%	0.0	0.0
5.	Soap (qualitative analysis)		–	
6.	Mass fraction of unsaponifiable matter	%	0.63	0.87
7.	Peroxide number	mmol active O/kg	3.2	5.7
8.	Flash point	°C	–	237.0
9.	Presence of solvent (gasoline) in oil (qualitative analysis)		–	+

Table 2. *Physicochemical parameters of cottonseed oil refined on adsorbent imported from Switzerland*

No.	Indicator name	Unit of measurement	Whitening soil consumption, %				
			1.0	1.5	2.0	2.5	3.0
	Refined, unbleached cottonseed oil obtained by pressing						
1.	Color, constant 35 yellow units:	red unit	9.4	6.0	4.6	3.8	3.5
		blue unit	0.6	0.2	0.0	0.0	0.0
2.	Acid number	mg KOH/g	0.24	0.24	0.23	0.22	0.23
3.	Mass fraction of moisture and volatile matter	%	0.06	0.06	0.05	0.05	0.05
4.	Mass fraction of unsaponifiable matter	%	0.61	0.58	0.55	0.51	0.48
5.	Peroxide number	mmol active O/kg	3.1	2.9	2.8	2.8	2.7
6.	Oil holding capacity of bleaching earth	°C	23.2	28.7	25.1	23.9	25.7
7.	Oil yield of bleached oil	%	99.74	99.54	99.47	99.38	99.18
	Refined, unbleached cottonseed oil obtained by extraction						
1.	Color, constant 35 yellow units:	red unit	14.7	12.2	10.3	8.6	6.8
		blue unit	1.5	1.0	0.6	0.3	0.0
2.	Acid number	mg KOH/g	0.31	0.31	0.32	0.30	0.31
3.	Mass fraction of moisture and volatile matter	%	0.07	0.06	0.06	0.06	0.05

No.	Indicator name	Unit of measurement	Whitening soil consumption, %				
			1.0	1.5	2.0	2.5	3.0
4.	Mass fraction of unsaponifiable matter	%	0.81	0.79	0.77	0.72	0.67
5.	Peroxide number	mmol active O/kg	5.5	5.5	5.4	5.4	5.3
6.	Flash point	°C	238.0	237.0	239.0	241.0	243.0
7.	Oil holding capacity of bleaching earth	%	25.8	24.3	26.7	25.6	23.7
8.	Oil yield of bleached oil	%	99.70	99.59	99.41	99.30	99.25

The oil capacity of the adsorbent varied between 23–28%. The oil recovery decreased with increasing bleaching earth content. This indicates a technological compromise, i.e., color improvement is achieved at the expense of increased adsorbent consumption, but excess adsorbent is not economically feasible.

Table 3. *Physicochemical parameters of cottonseed oil refined on adsorbent imported from China*

No.	Indicator name	Unit of measurement	Whitening soil consumption, %				
			1.0	1.5	2.0	2.5	3.0
	Refined, unbleached cottonseed oil obtained by pressing						
1.	Color, constant 35 yellow units:	red unit	10.1	7.8	6.2	5.1	4.7
		blue unit	0.7	0.4	0.2	0.1	0.0
2.	Acid number	mg KOH/g	0.24	0.24	0.23	0.23	0.23
3.	Mass fraction of moisture and volatile matter	%	0.06	0.06	0.05	0.05	0.05
4.	Mass fraction of unsaponifiable matter	%	0.63	0.60	0.57	0.54	0.52
5.	Peroxide number	mmol active O/kg	3.2	3.1	3.0	2.9	2.9
6.	Oil holding capacity of bleaching earth	°C	36.4	39.1	40.3	39.8	38.9
7.	Oil yield of bleached oil	%	99.61	99.39	99.17	98.98	98.81
	Refined, unbleached cottonseed oil obtained by extraction						
1.	Color, constant 35 yellow units:	red unit	15.3	13.1	11.4	9.9	8.6
		blue unit	1.6	1.2	0.9	0.5	0.2
2.	Acid number	mg KOH/g	0.31	0.31	0.31	0.30	0.30
3.	Mass fraction of moisture and volatile matter	%	0.07	0.06	0.06	0.05	0.05
4.	Mass fraction of unsaponifiable matter	%	0.83	0.80	0.78	0.75	0.74
5.	Peroxide number	mmol active O/kg	5.6	5.5	5.4	5.3	5.3
6.	Flash point	°C	239.0	239.0	241.0	240.0	242.0
7.	Oil holding capacity of bleaching earth	%	37.9	38.4	41.2	40.5	39.7
8.	Oil yield of bleached oil	%	99.58	99.37	99.13	98.94	98.75

Discussion and Conclusion. As can be seen from Table 1, it was found that the physicochemical parameters of refined, unbleached cottonseed oils obtained by pressing and extraction methods as the initial raw materials differ significantly. The color index of the oil obtained by the extraction method is higher (20.0 red and 2.8 blue units), which indicates that pigments and accompanying substances are preserved in a greater amount compared to the press oil (12.5 and 1.3). This is explained by the fact that during the extraction process with the help of a solvent, coloring pigments pass more into the extraction oil.

The fact that the peroxide value reaches 5.7 mmol active O/kg in the oil obtained by the extraction method is higher than that obtained by the press method (3.2), indicating a relatively intensive course of oxidation processes during the extraction process. Also, the content of unsaponifiables is higher in the extraction oil (0.87%), which indicates a more complex colloidal composition of the oil.

Therefore, the oil obtained by the extraction method is a more complex object for the bleaching process and creates a higher load at the adsorption stage.

Table 2 shows that the quality indicators of cottonseed oil bleached with different amounts (1.0–3.0%) of adsorbent produced in Switzerland showed a significant change in a number of results with an increase in the adsorbent dose.

When bleaching oil obtained by the press method, when the amount of adsorbent was increased from 1.0 to 3.0%, the red unit decreased from 9.4 to 3.5, that is, by approximately 62–63%. The blue unit decreased from 0.6 to 0.0. This decrease in color intensity is explained by the effective binding of pigment molecules on the surface of the adsorbent. However, in the range after 2.0%, the rate of color loss slowed down sharply (4.6→3.8→3.5). This indicates that an excess amount of adsorbent does not have a positive effect on the efficiency of the process.

A similar trend was observed in the oil obtained by extraction: the red unit decreased from 14.7 to 6.8 (≈54%). However, the final color value remained higher than that of the pressed oil. This is explained by the relatively high content of pigments in the oil.

The acid value of the oil obtained by the pressing method decreased from 0.24 to 0.22–0.23 mg KOH/g. The change was minimal, confirming that adsorption bleaching does not significantly affect free fatty acids. In the oil obtained by the extraction method, almost no change was observed. This result indicates that the bleaching process is mainly directed towards pigments and oxidation products, and the acid value decreases mainly at the neutralization stage.

Unsaponifiable in the oil obtained by the pressing method decreased from 0.61 to 0.48% (≈21%). In the oil obtained by the extraction method, it decreased from 0.81 to 0.67% (≈17%). This indicator indicates the ability of the adsorbent to selectively capture sterols, pigments and other high-molecular components.

The peroxide value in the oil obtained by the pressing method decreased from 3.1 to 2.7 mmol active O/kg. In the oil obtained by the extraction method, a decrease from 5.5 to 5.3 mmol of active O/kg was observed. The decrease in peroxide value is associated with the partial binding of hydroperoxide and secondary oxidation products by the adsorbent. However, the degree of reduction is limited, indicating that the bleaching process does not completely eliminate the peroxide value.

From the results in Table 3, it can be seen that when the amount of adsorbent in the oil obtained by the press method was increased from 1.0 to 3.0%, the red unit decreased from 10.1 to 4.7 (≈53%), and the blue unit decreased from 0.7 to 0.0. The tendency of the color decrease indicates the intensive course of the adsorption process, but the dynamics of the decrease slows down significantly after 2.0% (6.2→ 5.1→ 4.7). This indicates that the active centers of the adsorbent surface are gradually saturated. In the oil obtained by the extraction method, the red unit also decreased from 15.3 to 8.6 (≈44%). However, the final color values remain higher than in the pressed oil.

The acid number remained in the range of 0.24–0.23 mg KOH/g in the oil obtained by the press method, and in the range of 0.31–0.30 mg KOH/g in the oil obtained by the extraction method. A slight decrease in the content of moisture and volatile substances (0.06–0.05%) is associated with the process temperature and vacuum conditions.

Unsaponifiables in the oil obtained by the press method decreased from 0.63 to 0.52% (≈17%). In the oil obtained by the extraction method, they decreased from 0.83 to 0.74% (≈11%). This result indicates the ability of the adsorbent to partially capture high-molecular components, but the selectivity is limited.

The peroxide value in the oil obtained by the press method decreased from 3.2 to 2.9 mmol active O/kg (≈9%), and in the oil obtained by the extraction method decreased from 5.6 to 5.3 mmol active O/kg (≈5%).

The oil capacity of the Chinese adsorbent was recorded at higher values (36–40% in the oil obtained by the press method, 38–41% in the oil obtained by the extraction method). This indicates that the adsorbent retains more oil. As a result, the yield of bleached oil decreased from 99.61 to 98.81% (press) and from 99.58 to 98.75% (extraction).

In conclusion, the analysis shows that both adsorbents lead to a decrease in color and some accompanying substances, but their adsorption activity and selectivity are different. The Swiss adsorbent is characterized by high efficiency and lower oil loss, while the Chinese adsorbent is characterized by relatively low selectivity and high oil capacity. This indicates that the use of the Swiss adsorbent is acceptable. However, the difference in the cost of these two types of adsorbent requires the analysis of additional economic indicators when choosing them.

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