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Modelling and evaluating the batch deodorization of sunflower oil

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ABSTRACT

An engineering approach for evaluating the oil temperature during the batch deodorization is proposed. This approach employs a quasi-steady model including equations of steady-state heat transfer for consecutive time steps of computation. A new index – De-value – for assessing the efficacy of batch deodorization is proposed. The De-value represents the reduction of a key volatile component. It was established that the oleic acid is better to be used as a key component for assessing the deodorization of high oleic sunflower oil conducted at low temperature conditions – below 200 °C. The deodorization process of sunflower oil may be accepted as efficient if the De-value reaches a value 2 ± 0.2. The engineering approach proposed for evaluating De-value can be used as a tools for: (i) estimating the efficacy of process conditions applied at present; (ii) specifying eligible values of process parameters when a new process design will be established.

1. Introduction

Deodorization is a crucial refining stage with an important effect on the quality of oil refined. Principally, it is a steam distillation under vacuum. The purpose of this process is the removal of undesired volatile odoriferous components in vegetable oils, namely aldehydes, ketones, carbohydrates, and free fatty acids. Among them the heavier free fatty acids have the lowest vapor pressure and therefore they are the least volatile components (Bockisch, 1998). The saturated stearic acid $C_{18:0}$ with a molecular mass of 284.5 Da, the saturated palmitic acid $C_{16:0}$ with a molecular mass of 256.4 Da, the unsaturated oleic acid $C_{18:1}$ with a molecular mass of 282.4 Da and linoleic acid $C_{18:2}$ with a molecular mass of 280.4 Da are of the biggest interest because their concentrations in crude sunflower oil and in the oils before deodorization are the highest (Vasileva, 2003) and their vapor pressures are the lowest. The deodorized oils can be considered as a binary mixture including triglycerides (non-volatile in practice) and a key component – a volatile free fatty acid (Bockisch, 1998). The free stearic acid is used as a key component in the cases when the deodorization process is carried out at temperatures exceeding 200 °C (Vasileva, 2003). It is not found any criterion or an index cited in the specialized literature for assessing the reduction of undesired volatile components of the oil when the deodorization is carried out under variable temperature conditions.

At present, three types of deodorizers – batch, semi-continuous and continuous – are used in the practice. Semi-continuous and continuous deodorizers are best suited for large plants. These deodorizers enable the residence time of oil and the consumption of stripping steam to be reduced (Brekke, 1980). They can gain a heat recovery up to 50% and 85%, respectively (Carson, 1988). The batch deodorizers are more suitable for small plants because of their flexibility and very low instatement costs (Bockisch, 1998). A large part of Bulgarian refineries are small and medium enterprises and for that reason the batch deodorizers are widespread. The temperature of the oil in these batch deodorizers is variable throughout the process and it is low due to the technical background of these small refineries. Two stages can be specified during the batch deodorization: (i) indirect heating up to temperatures 145–160 °C, and (ii) indirect heating up to temperatures 175–210 °C under high vacuum conditions (with a residual pressure 0,3…1…15 kPa) and a simultaneous injection of stripping superheated steam.

The deodorization is a complex heat and mass transfer process. During deodorization some degradation reactions such as hydrolysis of triglycerides and *cis–trans* isomerization reactions of free fatty acids (Ceriani et al., 2008; Kemeny et al., 2001; Tasan and Demirci, 2003) take place as well. The equation of Bailey (1941) for the requirement of stripping steam is recognized all over the world (Bockisch, 1998; Brekke, 1980; Gavin, 1978; Leniger and Beverloo, 1975; Molchanov, 1965). The vaporization efficiency included in this equation can be determined more accurately if the approach presented by Ceriani and Meirelles (2005), Decap et al. (2004) and MacFarland et al. (1972) is taken into consideration. Dijksra (1999) showed that Bailey's equation is eligible for batch and crow-flow deodorizing systems but it is not suitable for evaluation of the stripping requirement of countercurrent continuous

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Nomenclature

a	mass concentration of the key volatile component – a free fatty acid (%)
a_I, a_F	Initial and final mass concentration of the key volatile component at the beginning and at the end of the process (%)
c, c_M	specific thermal capacity of the oil processed and the metal body of apparatuses, respectively (J kg^{-1} °C^{-1})
D	mass consumption of direct stripping steam (kg)
$\dot{D}$	mass flow of direct stripping steam (kg/s)
De	deodorization value (1)
De_1, De_2, De_3	deodorization values when the key component is stearic, oleic or palmitic acid, respectively (1)
Di	average absolute relative deviation between the temperatures of oil determined theoretically and experimentally (%)
F	heating surface of the apparatus (m^2)
k	overall coefficient of heat transfer (W m^{-2} °C^{-1})
m	number of the experimentally measured temperatures during the process
M, M_M	mass of the oil processed and the metal body of apparatuses, respectively (kg)
n	the total number of consecutive computing time steps (1)
p_A	absolute working/process pressure in the apparatus during the second stage (Pa)
p_S	the vapor pressure of a key component (Pa)
p_{S1}, p_{S2}, p_{S3}	the vapor pressure of stearic, oleic and palmitic acids, respectively (Pa)
T	temperature of oil processed (°C)
T_S	temperature of the heating medium – dry saturated steam (°C)
Δt	a short discrete interval of time used as a computing time step (s)
η	coefficient accounting the external heat losses (1)
μ	average molecular mass of triglycerides in sunflower oil (Da): $\mu = 880$ Da
ψ	vaporization efficiency (1): $\psi = 0.5,\ldots,0.8$

Subscripts

E, T	experimental and theoretical
i, i+1	related to the beginning and the end of a computing time step, respectively
1,2	related to the first and the second stage of deodorization process, respectively

deodorizing systems. He advanced a new equation for determining the stripping requirement of countercurrent deodorizers as the number of mass transfer units were taken into consideration. The mass transfer during batch and continuous deodorizers were simulated by Ceriani and Meirelles(2004b,c). They applied a differential distillation model as vapor–liquid equilibria of fat system (triacylglycerides, diacylglycerols, monoacylglycerols, free fatty acids, etc.) were described by group contribution equations for vapor pressures and activity coefficients. These excellent contributions applied a sophisticated set of equations with a large number (over 10) of empirical coefficients. So, these approaches are more suitable for scientific investigations and the process design of continuous deodorization processes.

The heat transfer during deodorization is also complicated and it is difficult to be modelled by more sophisticated techniques due to the following reasons: First, there are not sufficient and detailed investigations on the heat transfer in viscous liquids, such as oils, by means of a free convection in apparatuses without mechanical stirring. Second, there is a heat transfer in two-phase system including liquid oil and steam superheated during the second stage of the process.

The objectives of the present paper were to propose (i) a simplified engineering approach for predicting the oil temperature during batch deodorization; (ii) a dimensionless index for assessing the reduction of undesired components in vegetable oils; and (iii) an engineering approach for evaluating this index as the temperature history of the oil, the process pressure and the mass flow of stripping steam are taken into consideration.

2. Model of heat transfer

A quasi-steady energy balance model for simulating the heat transfer in vegetable oil was employed. The unsteady heat transfer was described by a sequence of short regular time intervals Δt (of the order of seconds). The heat transfer during each time interval was considered as steady. Moreover, the temperature field of the oil throughout apparatus' volume was assumed as uniform for each time interval.

The history of oil temperature was determined by means of a sequence of computing time steps which duration is equal to the time interval Δt. The oil temperature T_{i+1} at the end of each computing time step can be calculated by the following equation:

$$T_{i+1} = T_i + k \cdot F \cdot (T_S - T_i) \cdot \eta \cdot \Delta t / (M \cdot c + M_M \cdot c_M). \quad (1)$$

This equation was derived from the differential energy balance of the apparatus related to the beginning and the end of the computing step. It was assumed that the temperature T_S of the heating medium throughout the process is stationary. The temperature of the entire metal body with the steam coil/jacket was assumed to be equal to the temperature T_S of the heating medium for each computing step. It was taken into consideration that the intensity of heat transfer at steam condensation is high and the thermal conductivity of the metal body is high as well. The intensity of heat transfer was evaluated by the overall coefficient of heat transfer from the heating steam to the oil processed. Two values k_1 and k_2 of this coefficient for the first and the second stages, respectively, were adopted. The stripping steam used during the second stage leads to a reduction of heat transfer intensity. The lower value of the coefficient k_2 reflects this circumstance.

3. De-value

3.1. Definition

The De-value for evaluating the reduction degree of undesired odoriferous components in vegetable oils deodorized is defined by means of the following relation taking into account the reduction of a free fatty acid adopted as a key component

$$De = \frac{a_I}{a_F}. \quad (2)$$

3.2. Evaluation

The mass consumption D of stripping steam for a deodorization process conducted at a constant temperature T is determined by the following modified equation of Bailey (1941):

$$D = \frac{41 \cdot M \cdot p_A}{\mu \cdot \psi \cdot p_S} \lg \frac{a_I}{a_F}. \quad (3)$$

The change of concentration for a small interval of time Δt (in a range of seconds) can be expressed by means of the following equation derived from Eq. (3):

$$\lg \frac{a_i}{a_{i+1}} = \frac{\mu \cdot \psi \cdot p_S \cdot \dot{D}}{41 \cdot M \cdot P_A} \Delta t \quad (4)$$

The De-value for a process conducted at a variable oil temperature $T = f(t)$ can be determined by means of the following equation taking into account Eqs. (2) and (4):

$$\lg(\mathrm{De}) = \lg \frac{a_I}{a_F} = \sum_{i=1}^{n-1} \left(\lg \frac{a_i}{a_{i+1}} \right) = \sum_{i=1}^{n-1} \frac{\mu \cdot \psi \cdot p_S(T) \cdot \dot{D}}{41 \cdot M \cdot p_A} \Delta t \Bigg). \quad (5)$$

The duration t of a process carried out at a constant temperature T and ensuring a target De-value can be determined by the following equation derived from Eqs. (2) and (4):

$$t = \frac{41 \cdot M \cdot p_A \cdot \lg \mathrm{De}}{\mu \cdot \psi \cdot p_S(T) \cdot \dot{D}}. \quad (6)$$

4. Specific case study

4.1. Materials and methods

The process under study was carried out in a batch deodorizer (Fig. 1) representing a vertical cylindrical vessel with overall dimensions - diameter 2 m, height 4.1 m and a heating surface of the steam coils $F = 25\ \mathrm{m}^2$. The mass and the specific thermal capacity of the steel body with the steam coil were $M_M = 1900$ kg and $c_M = 0.46\ \mathrm{kJ\ kg^{-1}\ ^\circ C^{-1}}$, respectively. The specific thermal capacity of steel was read from Liley et al. (1999). The mass of the processed sunflower oil batch was $M = 6000$ kg. The specific thermal capacity of sunflower oil c as a function of the temperature T was determined by the correlation $c = 1700 + 2.1T$. The last fits to the experimental data presented in Liley et al. (1999), Molchanov (1965) and Souza et al. (2004). The pressure of the indirect heating steam and the corresponding temperature of the dry saturated steam were 1.05 MPa and $T_S = 182.1$ °C, respectively. The deodorization process was carried out under the following temperature conditions: the oil was heated from 80 to 145 °C during the first stage and from 145 to 178 °C during the second stage. Direct superheated steam with a mass flow $\dot{D} = 0.078$ kg/s 280 kg/h and temperature 250 °C was applied as a stripping agent. The absolute working pressure in the apparatus held during the second stage was equal to $p_A = 1$ kPa. The overall coefficient of heat transfer from the heating steam to processed oil was assumed to be equal to $k_1 = 100\ \mathrm{W\ m^{-2}\ ^\circ C^{-1}}$ and $k_2 = 65\ \mathrm{W\ m^{-2}\ ^\circ C^{-1}}$, respectively, for the first and the second stage. Their values were identified preliminarily on the basis of experimental oil temperature histories recorded in an apparatus with similar performance and temperature conditions. The identified values of these coefficients correspond to the minimum of the average absolute relative deviation Di (see Eq. (9)) between experimental and theoretical (Eq. (1)) temperature curves. The share of external heat losses was assumed to be equal to 3% corresponding to = 0.97. The vaporization efficiency was accepted $\psi = 0.6$. The heat transfer in the oil was simulated by the model proposed above as the computing time step was $\Delta t = 10$ s.

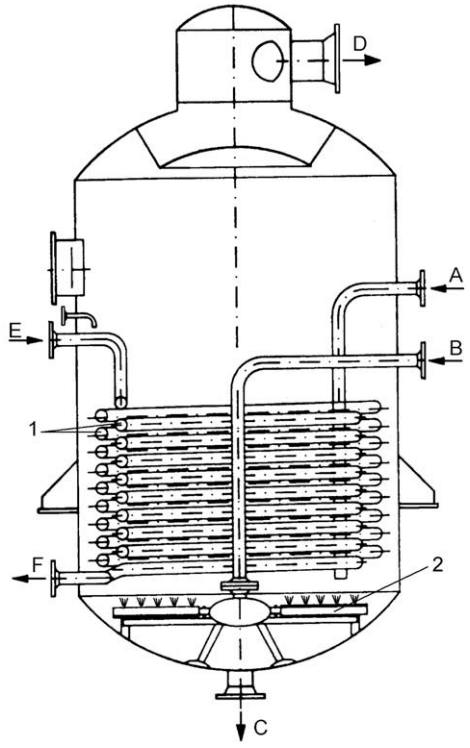

Fig. 1. Batch deodorizer: (1) internal heating pipe coils; (2) star-formed perforated pipe distributor; (A) neutralized bleached oil inlet; (B) stripping steam inlet; (C) deodorized oil outlet; (D) vapour outlet; (E) heating steam inlet; (F) condensate outlet.

The oil studied was derived from Bulgarian sunflower seeds of high oleic type. The initial mass concentration a_I of free fatty acids in sunflower oil before deodorization was assumed 0.1% as the concentrations of free stearic, palmitic, oleic, and linoleic acids were, respectively, 0.003%, 0.006%, 0.080%, and 0.011% according to Angelova (2006). The final mass concentration a_F of free fatty acids at the end of deodorization should be below 0.05% (Kellens and De Greyt, 2000), which corresponds to an acid value of 0,1 mg KOH/g according to the Bulgarian State Standard BDS 1–77 related to refined sunflower oil. The vapor pressure p_S of stearic, oleic and palmitic acids depending on temperature T were determined by the following Antoine's correlations (Vasileva and Pultsin, 1979):

$$p_{S1} = 133.10^{8.6874 - 3430.6/(T+221.1)} \quad (7a)$$

$$p_{S2} = 133.10^{7.9362 - 2566.4/(T+146.8)} \quad (7b)$$

$$p_{S3} = 133.10^{8.3019 - 3126.6/(T+222.9)}. \quad (7c)$$

These vapor pressures are in conformity with data presented in tables or charts in other sources – Brekke (1980), Fatty Acid Data Book (1992), Lide (2007) and Liley et al. (1999). The vapor pressures of these acids can be determined by means of the correlation of Ceriani and Meirelles (2004a) for a large number of fatty compounds. However this correlation includes 12 empiric coefficients. For that reason the last correlation is more suitable for fatty components for which there are no experimental data.

The sensitivity of process duration t from some process parameters used for process control (the temperature of oil T, the mass flow of stripping steam $\dot{D}$ and the working pressure p_A) was evaluated by means of the following functions of sensitivity (Tomovitch and Vukabrovitch, 1982):

$$\Phi_T = \frac{T}{t} \cdot \frac{\partial t}{\partial T} \approx \frac{T}{t} \cdot \frac{\Delta t}{\Delta T}; \quad \Phi_D = \frac{\dot{D}}{t} \cdot \frac{\partial t}{\partial \dot{D}} \approx \frac{\dot{D}}{t} \cdot \frac{\Delta t}{\Delta \dot{D}};$$
$$\Phi_P = \frac{p_A}{t} \cdot \frac{\partial t}{\partial p_A} \approx \frac{p_A}{t} \cdot \frac{\Delta t}{\Delta p_A}. \quad (8)$$

The applicability and accuracy of the quasi-steady model was evaluated by means of the average absolute relative deviation Di

(%) between the temperatures of the oil determined theoretically (T_T) and experimentally (T_E)

$$Di = \frac{1}{m} \cdot \sum_{1}^{m} \frac{|T_T - T_E|}{T_E} 100. \quad (9)$$

According to Grubov (1971) the applied approach can meet the engineering requirements when the average relative deviation Di is less than 10%. This index was used by Ceriani et al. (2008) and Ceriani and Meirelles (2004a), as well.

4.2. Results and discussion

The temperature history of oil simulated by the proposed model is presented in Fig. 2. The end of the first phase is pointed with an arrow. According to the figure, the total duration of the process was 397.2 min → 6.62 h, as the duration of the first and the second stages were 87.3 min and 300.6 min, respectively. If the mass of the metal body and the external heat losses are not taken into consideration the total process duration will be 359 min. If these two last factors are ignored, an error of 9.6% will be registered.

The experimental data of the vapor pressures of stearic, oleic and palmitic acids according to Liley et al. (1999) were compared together. It was ascertained that the vapor pressure of oleic acid is least of all when the temperature is up to 200 °C and the pressure of stearic acid is least of all when the temperature is over 200 °C. Therefore, the oleic and stearic acids are the least volatile components during a process carried out at temperatures up to 200 °C and over 200 °C, respectively.

The change of De-values during the process studied is presented in Fig. 3. The final values are as follows: $De_1 = 2.11$, $De_2 = 1.86$ and $De_3 = 7.33$ for the reduction of stearic, oleic and palmitic acids, respectively. Therefore, the final concentrations of stearic, palmitic, oleic and linoleic acids will be 0.0014%, 0.0008%, 0.0430% and 0.0059%, respectively. The reduction of linoleic acid was calculated by means of De_2-value because the molecular mass and, respectively, the vapor pressure of oleic and linoleic acids are very close. The corresponding total concentration of free fatty acids in the end of deodorization will be 0.051%. This means that the deodorization process studied assures the removal of undesired odoriferous components, as well as the removal of the least volatile free fatty acid to a sufficient extent. The total reduction of all free fatty acids obtained at the end of the process will be 0.1/0.051 = 1.96.

The duration of a process carried out at a constant temperature $T = 190$ °C and assured $De_2 = 2$ is 108.8 min according to Eq. (6). For this process $De_1 = 2.1$ and $De_3 = 6$. Therefore, the final concentrations of stearic, palmitic, oleic and linoleic acids will be 0.00144%, 0.0010%, 0.04% and 0.0055%, respectively. The corresponding total concentration of free fatty acids in the end of deodorization will be 0.0479%. The total reduction of all free fatty acids obtained at the end of the process will be 0.1/0.0479 = 2.09. For a process carried out at a constant temperature $T = 210$ °C and assured $De_2 = 2$ the process duration will be 40.7 min.

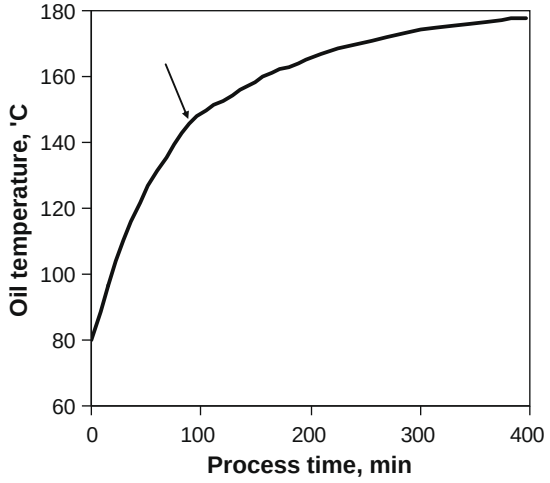

Fig. 2. Temperature history of oil processed.

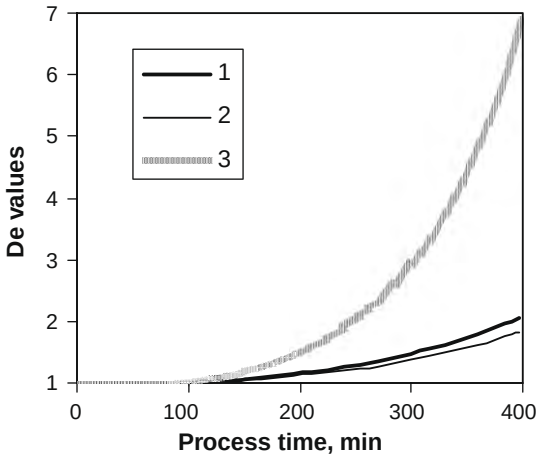

Fig. 3. Change of De-values related to stearic (1), oleic (2) and palmitic (3) acids.

In conclusion, the deodorization process under study was carried out in a temperature range up to 200 °C. The oleic acid is the least volatile component under those temperature conditions and its initial concentration is the highest in the processed high oleic sunflower oil. On the other hand, the De_2-value for oleic acid equal to 1.86 (Fig. 3), is close to the estimated reduction of all free fatty acids, which is 1.96. Therefore, the oleic acid could be used as a key component for assessing the batch deodorization of high oleic sunflower oil carried out at lower temperature conditions (below 200 °C).

The sensitivity of process duration t from some process parameters was evaluated by means of functions of sensitivity determined by Eqs. (6) and (8). Their values vary in the following ranges $\Phi_T = -9.9 \ldots -9.7$; $\Phi_D = -1$ and $\Phi_P = +1$. These functions of sensitivity show the percentage change of process duration t when 1% local deviation of oil temperature T, mass flow of steam $\dot{D}$ or working pressure p_A takes place, respectively. So, the increase of oil temperature with 1% leads to a decrease of process duration with 9.7–9.9% as the smaller value is related to higher temperatures. The process duration decreases exponentially when the temperature of oil rises. On the other hand, the process duration will be shortened by 1% when the mass flow of steam grows by 1% or the process pressure drops by 1%.

The approach proposed was applied successfully in the industrial practice of the refinery "Rosa" PLC–Popovo (Bulgaria). The efficacy of process conditions applied in the existing batch deodorizers were evaluated preliminary. The heating surface of apparatuses was enlarged, the other process parameters such as the temperature of oil at steam distillation, the process pressure and the mass flow of stripping steam were specified in order to minimize the total duration of deodorization. The eligible values of those process parameters were specified as the engineering background of refinery was taken into consideration.

Eight experimental temperature histories of oil were used for validating the quasi-steady model applied. It was found that the average relative deviations Di between the temperatures of oil determined theoretically and experimentally, as calculated by Eq. (9), were in the range of 7.3–9.7%. This validation shows the

accuracy of the approach proposed and its applicability in engineering practice. The quality of oil deodorized was appraised as good by means of a sensorial panel.

5. Conclusions

(1) An engineering approach for predicting the temperature history of oil during batch deodorization was proposed. This approach employs a quasi-steady energy balance model including equations of steady–state heat transfer for consecutive time steps of computation related to a short discrete interval of time. The approach was validated with experimental temperature histories.

(2) A new index – De-value – for assessing the efficiency of batch deodorization was advanced. The De-value expresses the reduction degree of a key volatile component in the oil. An engineering approach for its evaluating was also proposed.

(3) It was established that the oleic acid is better to be used as a key component for assessing the deodorization of high oleic sunflower oil conducted at lower temperature conditions (below 200 °C) in batch deodorizers.

(4) The deodorization process of high oleic sunflower oil may be accepted as efficient if the De-value related to the reduction of free oleic acid reaches a value 2 ± 0.2.

(5) The proposed approach can be applied for other kinds of oils, but their key component and the target De-value should be specified additionally.

(6) The duration of deodorization process may be reduced by 9.7–9.9% if the oil temperature is increased by 1%. Moreover, the duration will be reduced by 1% when the mass flow of stripping steam increases by 1% or the process pressure decreases by 1%.

(7) The De-value and the engineering approach for its evaluation were applied as a tool for:

- Estimating the efficacy of process conditions applied at present;
- Specifying eligible values of process parameters, such as process duration, temperature conditions, process pressure, mass flow of stripping steam and heating surface of apparatuses, when a new process design should be established.

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