Precise deodorization design for four different kinds of vegetable oils in industrial trial production

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Abstract

Trans fatty acids (TFA) have been shown to be associated with various health disorders. The major source of dietary TFA is high-temperature deodorization of vegetable oils. In this study, precision minimal deodorization was proposed to obtain healthier zero-TFA vegetable oils. Dual columns with dual temperatures (DCDT) deodorizer was designed, transformed and industrially implemented among dozens of plants. Also the deodorization temperatures were optimized and customized respectively for four kinds of vegetable oil (soybean oil and rapeseed oil: tray column 205°C and packed column 225°C, maize oil and sunflower seed oil: tray column 210°C and packed column 230°C). Four kinds of oils can all achieve zero-TFA by DCDT deodorization at the customized temperatures, and meanwhile oil qualities and shelf life stabilities were compared with corresponding conventional refining oils. The initial FFA and color were a little higher than conventional refining oils, however, they showed no significant differences in odors and shelf life stability, which indicated a good and stable oil quality of zero-TFA oils for future industrial productions and sales.

Keywords

TFA; deodorization temperature; dual columns with dual temperatures; oil quality

Abbreviations

TFA, trans fatty acids; FFA, free fatty acids; AV, acid value; PV, peroxide value; 3-MCPD, 3-monochloropropane-1,2-diol; GE, glycidyl esters; DCDT, dual columns with dual temperatures

Introduction

Vegetable oils, such as soybean oil, rapeseed oil, maize oil and sunflower seed oil are widely refined before being consumed or utilized as food supplements. The major steps of refining are degumming, neutralization, bleaching, and deodorization. Among these, deodorization is the final but the most critical step of refining as the final quality of vegetable oils is largely determined by the deodorization process. From an organoleptic point of view, oils should be light in color with a bland taste and a good oxidative stability. Both free fatty acids (FFA) and objectionable volatile compounds are removed from the oil by vacuum steam distillation at elevated temperatures. A fully refined oil contains low levels of FFA (usually$<$0.05%). Additionally, certain carotenoid pigments are destroyed resulting in a heat bleaching effect. However, micronutrients naturally occurring in oil such as tocopherol, sterol and squalene are partially removed during deodorization. Conventional deodorization temperature is usually 248-250°C or even higher, the heat treatment will induce a series of chemical transformations (oxidation reactions, cis-trans isomerization, cyclization, polymerization, etc.) due to the occurrence of numerous methylene-interrupted ethylenic double bonds (Fournier et al., 2006).

Several contaminants such as TFA, 3-MCPD and GE which are considered hazardous are generated during deodorization (Destaillats, 2012; MacMahon, 2014).

Nowadays, more and more attention is paid to the nutritional quality of vegetable oil. Oils should contain levels as low as possible for TFA, polymeric triglycerides, and secondary oxidation products and at the same time being rich in natural antioxidants. Therefore, alternative deodorization methods using milder conditions need to be developed to achieve these new quality requirements.

TFA in the human diet originate partly from natural sources, i.e. from meat and milk of ruminant animals (cows, sheep and goats), but the major part of human TFA exposure is the consequence of industrial food processing, like partially hydrogenation and deodorization of vegetable oils. Refined vegetable oils usually contain trans isomers of oleic acid (C18:1), linoleic acid (C18:2) and linolenic acid (C18:3), which may be formed during deodorization of oils rich in these unsaturated fatty acids (DeGreyt et al., 1996). Fully refined oils contain approximately 1-2% of TFA. There are several adverse biological effects of TFAs on body health. Various clinical studies and epidemiological observations showed that dietary TFA is associated with various health disorders such as diabetes, cardiovascular disease, obesity, breast cancer, prostatic cancer, infertility and coronary artery disease (Willett et al., 1993; Koletzko & Decsi, 1997; Hu et al., 1997; Lopez-Garcia et al., 2005; Micha & Mozaffarian, 2009; EL-Aal et al., 2019; Islam et al., 2019; Shah & Thadani, 2019). Guidelines for restriction or elimination of TFA were proposed around the world since the 1990s (Downs et al., 2013). The World Health Organization (WHO) called for the elimination of TFAs from the global food supply (Uauy et al., 2009). WHO's European Food and Nutrition Action Plan 2015-2020 suggested that TFAs should be less than 1% of the daily energy intake including TFA from natural origin. In 2018, WHO further proposed to eliminate industrially-produced trans fat from national food supplies, with the goal of global elimination by 2023. Chinese dietary reference intake guidelines (2013) suggested that TFAs from industrial origins should be less than 1% of the daily energy intake. In addition, according to the Chinese National Food Safety Standard for nutrition labeling of prepackaged foods (GB28050-2011) foods that have no more than 0.3 g TFA/100 g (solid) or 100 ml (liquid) can be labeled as having no TFA and can be called zero-TFA.

Many factors affect the overall quality of the oil obtained during the deodorization process, such as the structure of the deodorizer, deodorization temperature, deodorization time, vacuum and stripping steam. Therefore, the objectives of this work are to firstly design and transform a new deodorization system for vegetable oil using precision minimal processing, and then systematically evaluate the effect of this deodorization system on TFA content and soybean oil quality, as well as to provide an industrial alternative for the replacement of conventional high-temperature deodorization to obtain zero-TFA vegetable oil products.

Experimental Procedures

Materials

Degummed, neutralized and bleached soybean oils, rapeseed oils, maize oils and sunflower seed oils were used for this study. Crude oils were obtained from solvent extracted or screw pressed oil from Wilmar Group, and the crude oils conformed the internal standards of TFA[?]0.10% were selected. All chemicals used in this study for analyses were of analytical grade and purchased from Sigma-Aldrich (St. Louis, MO, USA).

Conventional deodorization

Neutralized and bleached oils were deodorized in an industrial deodorizer soft column, which was designed by Alfa Laval and has been widely used in industrial vegetable oil refining in recent years. The incoming bleached oils were firstly deaerated and then heated by heat exchanger and high-pressure steam to the final operating temperature of 248-250degC under reduced pressure (<2 mbar). Steam was applied as the stripping gas (approximately 1% of the oil weight). From the final heater the oils flowed by gravity into the deodorizer, through the deodorizer and out to be cooled by heat exchange with incoming bleaching oil. During flow through the deodorizer, the oil firstly underwent a continuous stage for fast FFA removal in a structured packing unit for a short time (5-10 min), and then a long time (90-120 min) holding stage for heat bleaching and deodorization.

Optimization of deodorization temperature

Deodorization temperatures were preliminarily optimized between 220-240degC in the soft column or tray column, in order to investigate the relationship between temperatures and TFA generation. Meanwhile deodorization temperatures were optimized between 225-235degC in a packed column, to investigate the relationship between temperatures and TFA generation. Other parameters (deodorization time, vacuum, stripping steam etc.) were not simultaneously adjusted as variable and remained at their best according to the actual conditions in each plant, to obtain deodorized oils with low FFA, light color and few odors.

Dual columns with dual temperatures deodorization

A new DCDT deodorizer was designed, upgraded and implemented in a large number of plants of Wilmar Group. This new deodorizer contained dual independent columns (tray column and packed column). Some plants combined independent tray column and packed column together, and some others firstly separated the soft column into a packed column and tray column, and then strung them in order (Fig. 1). During vacuum deodorization, oil from bleaching was heated by heat exchange with oil out from packed column to a moderate temperature (205degC for soybean oil and rapeseed oil, 210degC for maize oil and sunflower seed oil), after which it first passed through the tray column to undergo heat bleaching and deodorization for a long time (60-90 min). When the oil came out from the tray column, it was heated again by high-pressure steam to a higher temperature (225degC for soybean oil and rapeseed oil, 230degC for maize oil and sunflower seed oil) and then passed through the packed column for a short time (5-10 min) to undergo final deacidification and deodorization. The deodorized oil was then cooled by heat exchange with bleached oil to below 60degC.

Trans fatty acids analysis

TFA content was quantitatively analyzed by gas chromatography (GC) according to the national standard of China (GB 5009.257-2016).

Free fatty acids and acid value determination

Free fatty acids contents of oils were calculated as acid value divided by 1.99 according to the AOCS Official Method 940.28, by titration. Acid values were determined according to the AOCS Official Methods Cd 3d-63.

Peroxide values determination

Peroxide values were determined according to the AOCS Official Methods Cd 8-53.

COLOR analysis

Lovibond color was measured using Lovibond Tintometer (Model F, Lovibond, England) according to the ISO method 15305:1998. The R value was calculated as follows: R= (R+Y/10)/2, where R and Y represent the red unit and yellow unit, respectively. The type of cuvette used to determine the color has a path length of 133.4 mm.

Sensory evaluation

Ten trained panelists sniffed deodorized oil samples (150 g), which were filled in opaque bottles (220 ml) to avoid the influence of the color in the decision. These samples included the deodorized oils obtained at different deodorization conditions. Bottles were capped for more than 15 min prior to being tested. After smelling each sample, panelists were asked to score the samples according to the following four basic options of scale: “it does not smell”, “it smells, or seems to smell of refined oil”, “it slightly smells of undesirable odors”, and “it smells of undesirable odors” (coded with numbers from 1 to 4, respectively).

1.10 Shelf life stability test

zero-TFA oils and corresponding conventional deodorization oils were respectively divided into 900 ml PET bottles (815 g oil/bottle), hermetically sealed, and stored on the shelf at room temperature (20-28degC) for 18 months. AV, PV, COLOR and sensory performance were monthly evaluated and compared.

1.11 Statistical analysis

All analyses were performed in triplicate, and the data expressed as the means +- standard deviation (SD). Statistical analysis was carried out using SPSS program version 19.0. The significant difference among the data was evaluated by One-way Variance (ANOVA) combined with Tukey's adjustment test. Significant differences were assessed at P [?] 0.05. Boxplots, line-charts and bar-charts were generated by using Origin 8.5 software.

Results and discussion

Optimization of deodorization parameters

Deodorization is usually the last stage of the refining process of edible oils and has a crucial impact on the refined oil quality. The current deodorization process has three main objectives: (1) stripping of volatile components such as FFA, valuable minor micronutrients (tocopherols, sterols, etc.) and contaminants (pesticides, light polycyclic aromatic hydrocarbons, etc.); (2) actual deodorization by removal of different off-flavors; (3) thermal destruction of pigments (so-called heat bleaching). Apart from the desired effects, some unwanted side-reactions such as the formation of trans fatty acids may also occur during deodorization. The effects of process conditions (temperature, time, pressure and stripping steam) on the standard quality parameters and the nutritional quality of the refined oils were well understood, so deodorizer design and process conditions have been optimized to ensure minimal formation of trans fatty acids, maximal removal of FFA & volatile contaminants and a controlled stripping of valuable micronutrients.

Deodorizer design and upgrade

Conceptually, there are usually two types of technologies involved in the design and construction of deodorizers: columns with trays, in which different type of internal devices (special steam lift pumps, steam bubbling rings, etc.) are used in the tray to inject the stripping steam, and columns with packings. Here, mostly structured packings are used, due to its low-pressure drop of vapor phase and optimal gas-liquid contact (Shahidi, 2005). Both stripping column designs attempt to provide the best contact between the vapor phase and the liquid phase by creating a large contact surface, together with an optimal stripping steam distribution. Moreover, existing deodorizers have individual advantages. Tray columns can provide a certain holding period at the deodorization temperature for effective heat bleaching. However, the tray column still has certain defects. For example, the only one-time contact between oil and steam results in the usage of large amounts of direct steam and therefore low deodorization efficiency to remove FFA and flavor substances. Furthermore, the long time deodorization results in higher amounts of TFA. Certainly, the stripping efficiency can be improved by incorporating a packed column filled with structured packing, in which the contact surface between steam and oil is largely improved. Such a column has been designed by Alfa Laval (SoftColumn) and is widely used in industrial vegetable oil refining in recent years. Besides the continuous need for more efficient processes, new developments in deodorization technology are also driven by the increased attention to the nutritional quality of vegetable oil. As a result, *De Smet* developed the *DUAL TEMP*$^{(c)}$deodorizer (De Greyt et al, 1999). This deodorizer consists of several trays. In the first tray, the incoming oil is heated with high-pressure steam or a thermal fluid to a moderate temperature (e.g. 230degC), after which it passes through the first deodorizing trays. In these trays, deacidification and deodorization takes place under mild conditions (moderate temperature/moderate stripping). After the first tray, the oil passes to a second heating tray, in which the oil is heated to a final and higher temperature (250degC). In the last tray, final stripping and heat bleaching occurs. Only low amounts of TFA were generated because the time at high temperature is restricted.

Therefore, in order to obtain a final oil product with good nutritional and standard physicochemical qualities, a dual columns with dual temperatures (DCDT) deodorizer was for the first time proposed, designed and upgraded in this study. Negative temperature effects (geometrical cis-trans isomerization) can be maximally minimized by the use of DCDT deodorizers. The DCDT deodorizer was designed to operate at different temperatures to reach the best compromise between required residence time for deodorization and heat bleaching (at a low temperature) in a tray column and final stripping at a higher temperature (for a short period) in a packed column. The process flow was presented above.

Deodorization temperature optimization

Besides the design of the deodorizer, deodorization temperature is the most critical factor for oil quality among the four deodorization influencing parameters (temperature, time, pressure, stripping steam). A high temperature is beneficial for an efficient stripping of FFA and heat bleaching, but it also results in a higher loss of tocopherols and sterols (Hamn et al., 2013). More importantly, a high deodorization temperature is the most critical parameter for TFA generation. Reported activation energy values for the thermal cis-trans-isomerization of linoleic and linolenic acid are rather low, which is an indication that TFA are relatively easily formed at an elevated deodorization temperature (Shahidi, 2005). The degree of reaction will vary with the degree of fatty acid polyunsaturation. The higher the degree of polyunsaturation, the greater the tendency of the cis-acid to trans-acid formation. Different studies showed that the relative isomerization rate can be expressed as follows: C18:3(100)[?]C18:2(10)[?]C18:1(1) (Henon et al., 1999; Leon-Camacho et al., 2001). Consequently, oils with a relative high linolenic acid content, such as soybean oil and rapeseed oil, are more sensitive to cis-trans-isomerization than maize oil and sunflower seed oil during deodorization. Generally, trans-formation is negligible below 220degC, whereas it becomes significant between 220degC and 240degC, and exponential above 240degC (Shahidi, 2005). Industrial deodorizations are usually operated at temperatures between 240degC and 260degC, and approximately 1-3% of TFA are found in full refined oils.

However, limited lab-scale deodorization cannot precisely simulate industrial deodorization. In order to confirm the industrial deodorization parameters for zero-TFA vegetable oil production with good quality, dozens of pilot trials were firstly carried out with soybean oil among plants to optimize deodorization temperatures of existing deodorizers (tray column, packed column and soft column). Meanwhile, the other parameters (deodorization time, vacuum, stripping steam etc.) were not simultaneously adjusted as variable and remained as usual at their best according to the actual conditions in each plant. Fig.2 shows results from the optimization of deodorization temperature in packed columns (Fig. 2a) or tray columns & soft columns (Fig.2b), and the relationship between TFA content and deodorization temperature. Since the oil would stay a long time in tray columns as well as in soft columns, the results were combined into Fig.2b. In contrast the oil would stay in packed columns for a much shorter time. The trial temperature was variable among 220-240degC. Approximately one-third of trial batches achieved TFA[?]0.3%. Interestingly, the majority of them were deodorized by packed column, while TFA were considerably more than 0.30% with the use of tray columns and soft columns, and increased rapidly to an average of 1.34% when the temperature was increased to 240degC. Therefore, to achieve the goal of TFA[?]0.3%, the upper limit of deodorization temperature for packed columns and tray columns & soft columns were significantly different. The temperature for packed columns was suggested to be no more than 235degC, while the temperature for tray columns & soft columns should be lower than 220degC. This might be due to the significant differences between deodorization times in columns with trays compared to packed columns.

The deodorization temperature was furthermore optimized in this new DCDT system using soybean oil. The results showed that temperatures for packed columns and tray columns should be controlled to a maximum of 225degC and 205degC respectively (Fig.3). However, the deodorization temperature cannot be infinitely low. The volatility of a given components (FFA, tocopherols, sterols, etc.) is expressed by its vapor pressure, which increases with increasing temperature. The lower the vapor pressure, the lower the volatility and, thus, it is more difficult to remove the components from the oil. For example, a moderate temperature deodorization was operated in neutralized red palm oil but found that FFA and all volatile components of odor could not be reduced although the oil was deodorized for 2 hours at 150degC (Riyadi et al., 2016). Therefore, to balance low TFA and oil physicochemical quality, the deodorization temperature for soybean oil in the DCDT system was calculated to be 225degC for packed columns and around 205degC for tray columns.

The temperatures for rapeseed oil, maize oil and sunflower seed oil were then respectively optimized and customized, which are shown in Table 1. Soybean oil and rapeseed oil have similar, but higher linolenic acid content (respectively 4.2-11% and 5-14%) than the other oils, so the temperatures for them were designed the same. Due to the lower content of linolenic acid in maize oil ([?]2%) and sunflower seed oil ([?]0.3%), the temperatures for these two oils can be set higher to further ensure the physicochemical quality of deodorized oils. Comparing the new zero-TFA oils with refined oils collected from the market ten years ago (internal unpublished data) it can be shown that TFA content decreased by more than 90% (Fig.4). Furthermore, comparing zero-TFA oils with currently available conventional refined oils an 80% decrease in TFA content is observed (Fig.5).

Refined oil quality

The quality of a refined oil is usually evaluated by traditional quality parameters such as a low residual FFA content, a light color, and a neutral odor and taste. In addition, high-quality vegetable oils should contain as low TFA levels as possible, high amounts of micronutrients, low levels of polymeric and oxidized triacylglycerols, and no contaminants (polycyclic aromatic hydrocarbons, etc.). Therefore, zero-TFA oils produced by DCDT deodorization were tested for physiochemical quality and shelf-life stability.

FFA

The efficiency of deodorization is usually quantified by the reduction of FFA. For most conventional-processed 1^{st}-grade refined oils, the target residual FFA content is 0.03-0.05%, and the upper limit for FFA in past national standards for these oils in China was 0.10%. In order to conform to the development trend of minimal processing, the national standards for these oils in China have been revised and updated successively in recent years, and the upper limit for FFA is currently 0.25%. Fig. 6 shows the comparisons of FFA of four oils (soybean oil, rapeseed oil, maize oil and sunflower seed oil) obtained by conventional high-temperature deodorization or DCDT deodorization. The content of FFA of these deodorized oils are all less than 0.08%, not only meeting the internal quality standard for the trial-production ([?]0.10%) but also meeting the national standards for 1^{st}-grade refined oils. Since FFA contents of oils obtained by the DCDT deodorization were all slightly higher than the conventional ones due to the lower deodorization temperatures, the oil stability during the shelf life might be a concern and will be discussed later.

Odor

A bland odor is a very important feature of refined oils. Experiences have shown that flavor and odor removal correlate well with FFA reduction. Removal of odoriferous compounds is similar to FFA stripping and mainly related to steam stripping. Nevertheless, some differences exist among different oils. Soybean oil and rapeseed oil have typical odors, which often require more steam treatment to remove. In our study, the stripping steams were all kept at optimum condition according to the actual situation in each deodorizer to ensure maximum stripping of FFA and odoriferous compounds. According to sensory evaluation results comparison shown in Fig. 7, there were no significant differences between the DCDT and conventional oils, both with bland odor and taste, without any off-flavors.

Color

Besides a bland odor and taste, a light color is also a quality parameter for most refined oils. In the refining process, the color components can be removed during the bleaching process by adsorption onto a suitable bleaching earth or by thermal degradation during deodorization. Heat bleaching is a purely time-temperature-dependent reaction. Literature data on heat bleaching of palm oil indicate that the rate of carotene breakdown doubles per 20degC increase in temperature and heat degradation of carotene takes a few hours at 210degC but only a few minutes at 270degC (Shahidi, 2005). Similarly, to conform to the trend of minimal processing, specific restrictions of R value have been revoked and replaced with descriptive words in the newest Chinese national standards for these refined oils.

Fig. 8 shows the comparisons of R-values of four oils (soybean oil, rapeseed oil, maize oil and sunflower seed oil) between conventional high-temperature deodorization and the DCDT deodorization. The majority of oils have an R-value below 1.5R. For DCDT deodorized oils the R-value of sunflower seed oil was the lowest ([?]1.0R), followed by soybean oil, rapeseed oil and maize oil. Except sunflower seed oil, R values of the other zero-TFA oils were all slightly higher than corresponding conventional ones.

Shelf life stability

Although the initial value of quality parameters (FFA and COLOR) of zero-TFA deodorized oils were a modestly higher than conventional deodorized oils, there were no significant differences in the shelf life stability between the oils processed by these two technologies. Soybean oil is shown as an example in Fig.9. Changes during the 18-month shelf life of important quality parameters (AV, PV, COLOR) for conventional refined oils and "zero TFA" oils are illustrated. The AV of both soybean oils increased slightly during the whole shelf life and well met the national standard for 1^{st}-grade soybean oil ([?]0.5 mgKOH/g), which means that there was no significant difference in AV change trends between the different processed oils. The PV is an important indicator for the degree of primary oxidation of fats and oils. Hydroperoxides can be decomposed to volatile and non-volatile lower molecular weight components such as aldehyde and ketone at temperatures above 100degC (Tsiadi et al., 2001) used during the deodorization and therefore the PV value of freshly finished oils were all zero. During the whole shelf life, it was found that both soybean oils had a rising trend in PV, and most of the time conventional rerfined soybean oil showed higher PV than zero-TFA soybean oil. The color (R-value) of these two soybean oils both rose to a certain extent (Fig.9), but at end of the 18-month-shelf live there was no differences between the two oils. Besides, the flavor of them did not show significant differences in changes during the shelf-life either (data not shown). Not only soybean oil, but also rapeseed oil, maize oil and sunflower seed oil showed similar trends in these quality parameters during the shelf-life test obtained by the two different deodorization methods (data not shown). In conclusion, DCDT and conventional deodorization of several kind of oils had similar overall shelf-life stability without significant differences, which is critically important for industrial vegetable oil production and sales.

Conclusions

Deodorization is a key step during oil refining for overall oil quality, especially the deodorization temperature is the direct and critical parameter influencing TFA generation. A minimal deodorization system DCDT was developed, transformed and implemented among dozens of plants at Wilmar Group. TFA content of vegetable oils (soybean oil, rapeseed oil, maize oil and sunflower seed oil) were all significantly reduced to below 0.3%, which can be called zero-TFA in China. Furthermore, oil quality including physiochemical and oxidative quality did not show significant differences between zero-TFA oils and conventional refining oils. Nowadays, these zero-TFA oils have been scale produced and supplied to consumers. Furthermore, more studies will be needed to investigate other parameters such as deodorization times, vacuum pressure and stripping steam in the deodorization which might also be important for oil quality, especially the retention of micronutrients (tocopherols, plant sterols, etc.).

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Conflict of interest

The authors declare that they have no conflict of interest.

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Tables

Table 1 Dual temperatures used for soybean oil, rapeseed oil, maize oil and sunflower seed oil deodorization

	Tray column (°C)	Packed column (°C)
Soybean oil	205	225
Rapeseed oil	205	225
Maize oil	210	230
Sunflower seed oil	210	230

Figure legends

Fig.1 Dual columns with dual temperatures (DCDT) deodorizer

Fig.2 The relationship between TFA generation in soybean oil and temperatures of packed column (a), tray column and soft column (b) in conventional deodorization system

Fig.3 The relationship between TFA generation in soybean oil and temperatures of packed column (a) and tray column (b) in DCDT system

Fig. 4 Comparison of TFA between refined oils in 2008 and zero-TFA oils in 2019

Fig. 5 Comparison of TFA between conventional refined oils and zero-TFA oils

Fig. 6 Comparison of FFA between conventional refined oils and zero-TFA oils

Fig. 7 Comparison of flavor between conventional refined oils and zero-TFA oils

Fig. 8 Comparison of color between conventional refined oils and zero-TFA oils

Fig.9 Physiochemical quality parameters changes during 18-month shelf life of conventional refined and zero-TFA soybean oil (a: acid value, b: peroxide value, c: R value of color)

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