

# Analysis of Physicochemical Properties and Antioxidant Capacity of Evening Primrose Oil during Refining

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**Abstract:** Evening primrose oil is a rare oil, which is rich in linoleic acid, palmitic acid, oleic acid and linolenic acid and many other unsaturated fatty acids that are beneficial to human body. It has extremely high nutritional value and health care function, so it can play a role in the inhibition and prevention of some diseases. This topic proposed by means of evening primrose oil in the refining process, the sample of different refining link to the physical and chemical properties, volatile components and antioxidant capacity test and analysis, aims to explore the evening primrose oil, in the process of production of nutrient losses, and causes of its nutrient loss, to improve the quality of evening primrose oil, grease to provide scientific theory basis. The main research contents are as follows: (1)The national standard method was adopted to determine the main physicochemical indexes of evening primrose oil in different refining processes, and the changes were analyzed. The changes of fatty acid content and composition of evening primrose oil during refining were determined and analyzed. (2)DPPH, ABTS, FRAP and ORAC in vitro antioxidant evaluation methods were used to determine the changes of antioxidant capacity of evening primrose oil during the refining process, and Pearson bivariate correlation analysis was used to obtain the correlation between the contents of tocopherol, total phenol, phytosterol and squalene in evening primrose oil and antioxidant capacity.

**Keywords:** evening primrose oil, refining process, trace elements, antioxidant capacity

## 1. Introduction

Evening primrose, commonly known as noctiluca, mountain sesame, is a perennial herb. Domestic and foreign research shows that, due to the evening primrose oil is rich in unsaturated fatty acid and many beneficial to human body trace active ingredient, therefore, it has obvious characteristics of anti-inflammation, anti-oxidation, anti-thrombosis, lowering blood lipid, lowering blood sugar, losing weight, and also has significant curative effect on diabetes, hyperlipidemia, atherosclerosis, coronary heart disease and other diseases .

This project proposed by evening primrose oil in the refining process (degumming, deacidifying, decolorization, deodorization and dewaxing oil as raw material of samples, respectively, to determine its physical and chemical characteristics of each stage (acid value, peroxide value, uv absorbance, anisidine value, 2 - thio barbituric acid value, iodine value and saponification value), and in vitro antioxidant capacity were analyzed, and the change rule of its trace nutrients in the process of refining and explore the correlation between antioxidant capacity, aimed at for parameter optimization of evening primrose oil processing, To better maintain the nutritional value of evening primrose oil, improve its antioxidant activity to provide a theoretical basis for the better development and utilization of evening primrose oil.

## 2. Methods and Discussion

### 2.1. Acid value

The acid value of evening primrose oil was determined by referring to the third method in GB 5009.229 -- 2016, namely thermal ethanol indicator titration. In this experiment, the acid value of evening primrose oil after each process in refining was measured and the significance of its difference

was analyzed. See Figure 1 for comparison.

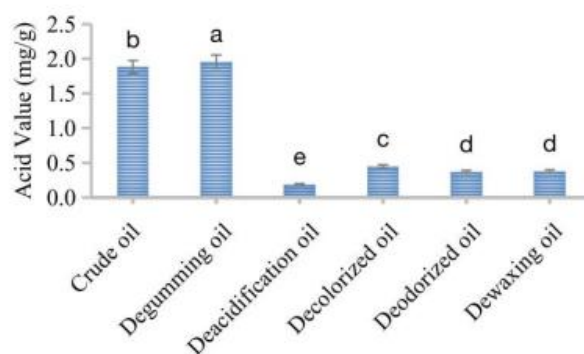


Figure 1: Changes in oleic acid price of evening primrose during refining

It can be seen from Figure 1 that after the deacidification process, the acid value of evening primrose oil changed significantly ( $P < 0.05$ ), decreasing from 1.84 mg KOH/g to 0.16 mg KOH/g. As part of the free fatty acids were neutralized by the addition of sodium hydroxide during the deacidification alkali smelting process, the acid value of evening primrose oil was significantly reduced. After the decolorization process, on the one hand, it may be the ester hydrolysis caused by the active clay; on the other hand, it may also be the adsorption of sodium ions by the adsorbents during decolorization to form free fatty acids, leading to a slight increase in the acid price of evening primrose oil to 0.47 mg KOH/g. Finally, after deodorization and dewaxing, the acid price dropped again to 0.34 mg KOH/g. The reason was that part of the free fatty acids and inorganic acids were taken away from the system by high-temperature water vapor in the deodorization process, while in the dewaxing process, the free fatty acids would crystallize at low temperature and be removed from the system.

## 2.2. Peroxide value

The determination method refers to the titration method in GB 5009.227-2016. The peroxide value is an important index to evaluate the degree of oxidative rancidity of vegetable oils and is also an index to characterize the content of primary oxidation products in vegetable oils. In general, the rancidity of vegetable oils is positively correlated with the peroxide value. The peroxide value of evening primrose oil in each refining process was determined and the significance of the difference was analyzed, as shown in Figure 2.

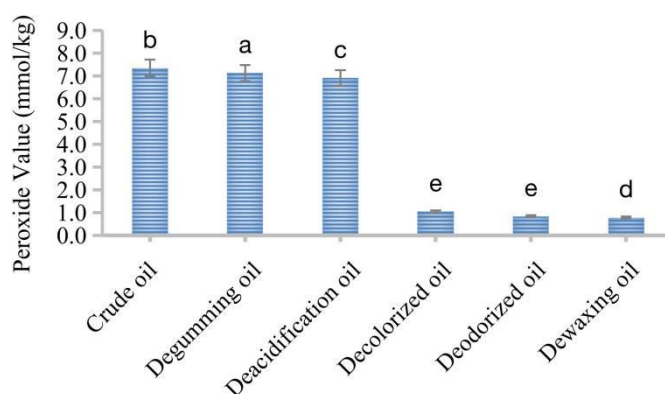


Figure 2: Changes in peroxide value of evening primrose oil during refining

As can be seen from Figure 2, the peroxide value of the hair oil of evening primrose before refining was as high as 7.33 mmol/kg. One possibility is that the antioxidant measures were not taken in time after the harvest of evening primrose seeds, leading to oxidative damage of evening primrose seeds. The peroxide value changed significantly after decolorization, from 6.89 mmol/kg to 1.01 mmol/kg. After analysis, part of the reason is that the adsorbent adsorbs the peroxide in evening primrose oil in the decolorization process. At the same time, due to the high temperature of decolorization process, part of the primary oxides in evening primrose oil are decomposed, resulting in a significant decrease in the peroxide value. The deodorization and dewaxing processes were carried out in a high temperature and vacuum environment. The peroxides may have volatilization and decomposition under this condition,

which further reduced the peroxide value to 0.77 mmol/kg. In conclusion, the peroxide value of evening primrose oil changed most significantly in the decolorization stage ( $P < 0.05$ ), so the decolorization stage had a significant effect on the quality of evening primrose oil, and the reasonable selection of adsorbents was crucial to the quality of oil. After refining, the peroxide value of evening primrose oil was less than 6.0 mmol/kg, which was in line with the standard of first-grade edible vegetable oil.

### 2.3. Ultraviolet absorbance

The determination method refers to GB/T 22500 -- 2008/ISO 3656:2002. Different fatty acid composition can show different absorption spectra in the ultraviolet spectrum region, and UV absorption spectra can be used to better evaluate the quality and oxidation degree of oils. According to the ultraviolet absorbance of evening primrose oil in each refining process, the value of conjugated diolefins and triolefins were determined to further evaluate the quality and oxidation degree of the oil. The analysis is shown in Figure 3.

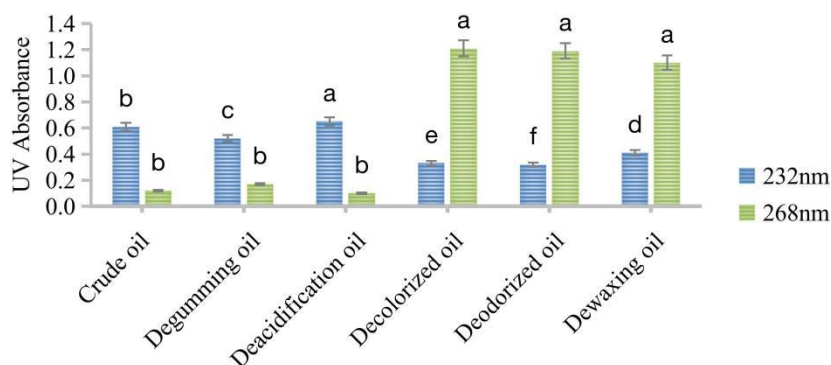


Figure 3: Changes in UV absorbance of evening primrose oil during refining

As shown in Figure 3, the UV absorbance (232 nm) of evening primrose oil decreased during the refining process, and it was the most obvious after the decolorization process, decreasing from 0.63 to 0.31, which was consistent with the changing trend of peroxide value, with a large decrease in both cases. The reason may be that the high temperature further decomposes the primary oxidation products in the oil. The value of conjugated diolefin increased slightly after dewaxing, but still decreased significantly compared with that before refining. The variation trend of conjugated triolefin value (268 nm) is just opposite to the conjugated diolefin value, and it is significantly increased in the decolorization stage, which may be because the further decomposition of primary oxidation products into secondary oxidation products leads to the rise of conjugated triolefin value.

### 2.4. Iodine value

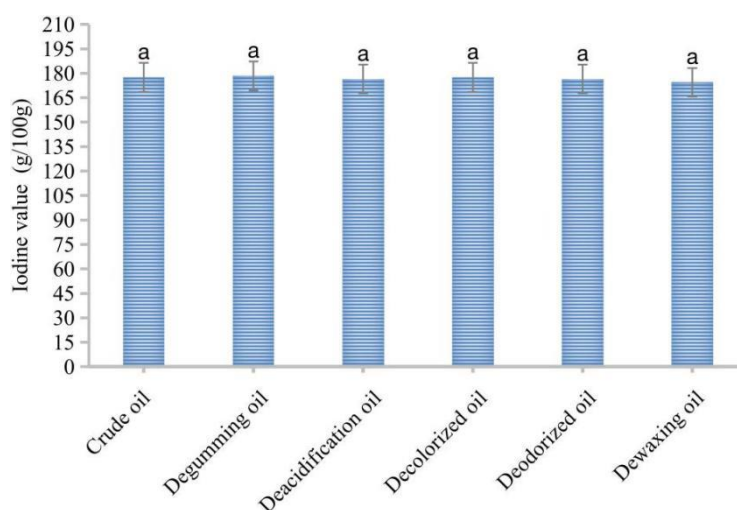


Figure 4: Changes in iodine value of evening primrose oil during refining

The determination method refers to GB/T 5532 -- 2008, and the iodine value can reflect the proportion of unsaturated fatty acids in fats. The content of unsaturated fatty acids is positively correlated with the oil's unsaturation, and the oil with high iodine value usually has a greater unsaturation. Therefore, the iodine value of evening primrose oil in each refining process was determined, and the analysis changes were shown in Figure 4.

The iodine value of evening primrose oil was 177.31 g/100g before refining, and its crude oil had a high degree of unsaturation. Meanwhile, as can be seen from the figure, its iodine value did not change significantly during the refining process ( $P > 0.05$ ), but remained at 173.23 g/100g after refining. Baoli Wen et al. analyzed the change of iodine value of Perilla seed oil during the refining process and concluded that its iodine value did not change significantly after refining. In conclusion, the iodine value of evening primrose oil does not significantly change after refining ( $P > 0.05$ ), and the trend of the iodine value obtained in this experiment during refining is similar to the existing conclusions.

### 2.5. Anisidine value

The determination method refers to GB/T 24304-2009 /ISO 6885:2006. Anisidine value can characterize the proportion of secondary products such as aldehydes and ketones in vegetable oil, and generally, the oxidation degree of oils is positively correlated with the content of aldehydes and ketones in oils. Therefore, the anisidine values of evening primrose oil in each refining stage were measured, and the analysis results were shown in Figure 5.

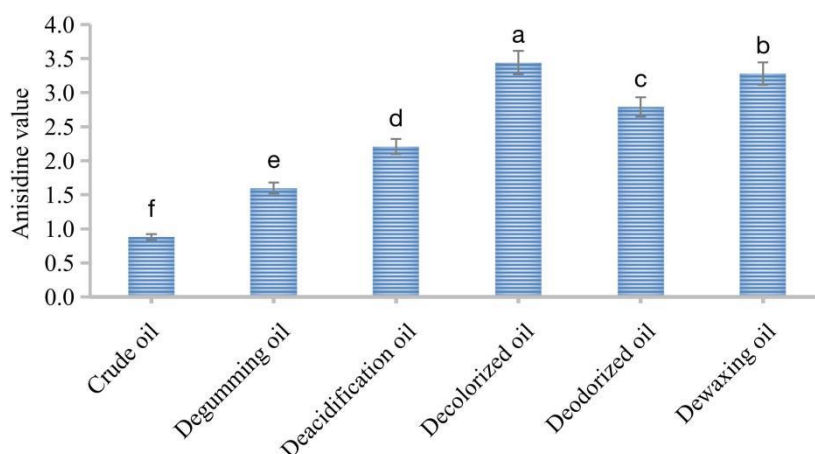


Figure 5: Changes in anisidine value of evening primrose oil during refining

In Figure 5, the anisidine value of evening primrose oil significantly increased from 0.86 to 3.27 in the refining process, and the most prominent increase of anisidine value was observed after the decolorization process ( $P < 0.05$ ), possibly because the catalytic effect of bleaching agent used in decolorization promoted the decomposition of hydroperoxides in the oil to generate a large number of secondary oxidation products. However, after the deodorization process, it is likely that the secondary oxidation products in evening primrose oil volatilized and degraded again, leading to the corresponding decrease of anisidine value. In conclusion, after refining, the anisidine value of evening primrose oil was significantly increased ( $P < 0.05$ ), and the effect of decolorization process was the most obvious.

### 2.6. Saponification value

The determination method refers to GB/T 5534 -- 2008, and the average molecular weight of all fatty acids is inseparable from the saponification value, which can measure the total amount of fatty acids in lipids. The saponification value can also indirectly reflect the composition of fatty acids and the specific gravity of glycerol. Therefore, saponification values of evening primrose in each refining process were measured and analyzed, and the results were shown in Figure 6.

As can be seen from the figure, saponification values of evening primrose oil did not change significantly during the refining process. Before refining, the saponification value was  $194.16 \pm 3.06$  mg KOH/g, which decreased slightly during degumming and deacidification. After refining, the saponification value was  $187.35 \pm 3.66$  mg KOH/g. Zhu Yukun et al. found that the refining process did not significantly change the saponification value of eucommia seed oil. In summary, the saponification value of evening primrose oil was measured and analyzed.

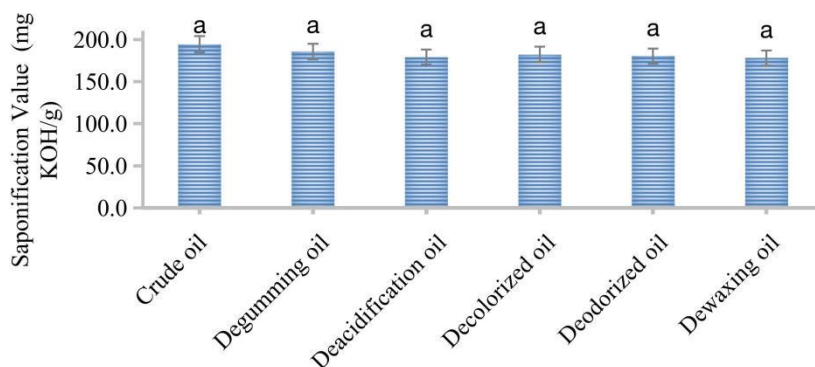


Figure 6: Changes in saponification value of evening primrose oil during refining

### 2.7. Thiobarbituric acid value

The determination method refers to GB/T 35252-2017. The 2-thiobarbituric acid value (TBA value) in vegetable oil can characterize the degree of lipid oxidation. By measuring its TBA value, the content changes of secondary oxidation products of evening primrose oil in each refining process can be further analyzed, as shown in Figure 7.

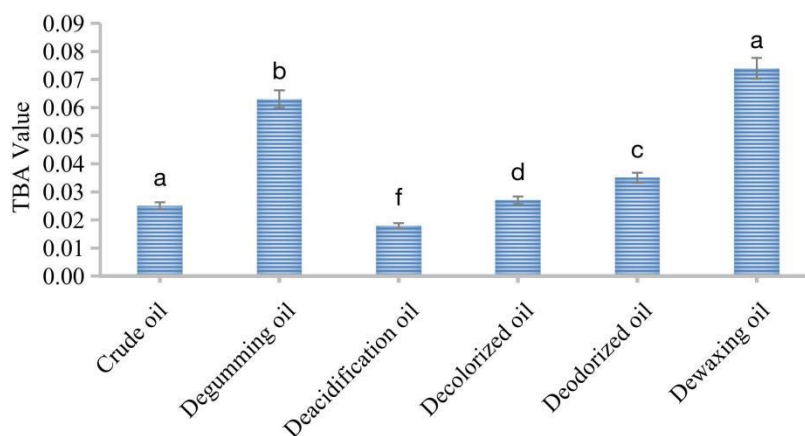


Figure 7: Changes in TBA values of evening primrose oil during refining

As can be seen from Figure 7, the 2-thiobarbituric acid value of evening primrose oil increased significantly after hydration degumming ( $P < 0.05$ ), but decreased significantly after alkali refining and deacidification ( $P < 0.05$ ). However, it still showed an upward trend during the whole refining process, and increased significantly in the decolorization and deodorization processes, probably because the secondary oxidation products in evening primrose oil increased under the high temperature conditions in the two refining processes. I

### 2.8. Changes in antioxidant capacity

The automatic oxidation of evening primrose oil during the refining process is easy to affect the quality of oil, and the automatic oxidation of vegetable oil starts from free radical reaction. Therefore, four *in vitro* antioxidant evaluation methods, DPPH, ABTS, FRAP and ORAC, were selected to comprehensively evaluate the antioxidant capacity of evening primrose oil during the refining process *in vitro*. In addition, the correlation between the micronutrient components (tocopherol, total phenol, phytosterol and squalene) in evening primrose oil and its antioxidant capacity was explored, and the effect of refining process on the quality of evening primrose oil was comprehensively analyzed.

## 3. Conclusion

By measuring the changes of fatty acid content, volatile components, basic physical and chemical indexes and antioxidant capacity of evening primrose oil in the refining process, and further analyzing the data, the following main conclusions were drawn.

(1) Through the detection of various physicochemical indexes of evening primrose oil in the refining stage, it can be found that with the refining process, the acid value and peroxide value of evening primrose oil decreased significantly, while the iodine value did not change significantly, indicating that the quality of evening primrose oil improved after refining, and the content of unsaturated fatty acids remained basically unchanged. Among them, the alkali refining and decolorization stages had the greatest effect on the acid value of evening primrose oil, the acid value of the alkali refining stage decreased significantly, and the decolorization stage increased significantly. Decolorization stage has the most obvious effect on the peroxide value. The UV absorbance of crude oil at 232 nm decreases after refining. The UV absorbance at 268 nm shows a certain upward trend, and the decolorization stage has the most obvious effect on it. The anisidine value showed an increasing trend overall, and the effect of decolorization stage was most significant. It can be seen that the decolorization stage is an important stage in the refining process of evening primrose oil, which has a great influence on the quality of evening primrose oil. The parameters of the decolorization stage should be strictly controlled to ensure the quality of evening primrose oil.

(2) Through the analysis of the changes in antioxidant capacity of evening primrose oil in the refining stage, it can be found that the antioxidant capacity of evening primrose oil in the refining process is generally decreasing, and after the alkali refining and deacidification process, the four kinds of antioxidant capacity of evening primrose oil all decreased the most significantly. So in the refining process is able to get rid of evening primrose oil is not a benefit to the body of the impurities and improve the quality and taste of oils and fats, but at the same time, also make the oil oxidation resistance reduced significantly, to control the process parameters in the process of refining provide certain theoretical basis for improving oil quality.

## References

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