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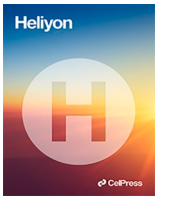

Research article

The development and application of compound formulation of natural antioxidants in *Idesia polycarpa* maxim. Oil

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ABSTRACT

Idesia polycarpa Maxim. is valued for its high oil yield, which fruit has high oil content and good health effects. However, the large amount of unsaturated fatty acids in the oil is easily oxidized, and its storage intolerance has seriously restricted its marketing. In order to slow down the oxidation rate of *Idesia polycarpa* Maxim. oil, this study found that the most effective single antioxidant was rosemary extract through the screening of seven different natural antioxidants. The experiments were designed using the response surface method to compound three individual natural antioxidants with good anti-oxidative effects, namely rosemary extract, tea polyphenol palmitate, and ascorbic acid palmitate. A comprehensive model was established to determine the optimal anti-oxidative compound formulation, which was found to be 0.45 g/kg of rosemary extract +0.21 g/kg of tea polyphenol palmitate. The effectiveness of the best compounded natural antioxidants under simulated frying temperature (160 °C) was then examined. The results showed that the antioxidant capacity of the compound formulation was significantly increased, and the generation of secondary oxidation products was inhibited and the loss of unsaturated fatty acids was reduced. Therefore, the compound antioxidant-added *Idesia polycarpa* Maxim. oil not only showed good storage resistance, but also improved the safety of use under high temperature environment. The results of this study significantly improved the oxidative stability of *Idesia polycarpa* Maxim. oil, provided a theoretical basis for replacing synthetic antioxidants, promoted the industrial development of *Idesia polycarpa* Maxim. oil and expanded other applications.

1. Introduction

Idesia polycarpa Maxim. is a deciduous tree belonging to the family of Flacourtiaceae, spanning the warm belt and subtropical zone in East Asia, and is mainly found in southern part of China at altitudes of 300~1200 m on the slopes of mountains, both sides of the sparse forest of valleys or forest edge [1]. It typically takes approximately 4–5 years for *Idesia polycarp*a to attain reproductive maturity and can serve as an exceptional garden tree species with high esthetic value and drought-tolerate [1,2]. Both the fruits and seeds of this

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species are capable of extracting oil [3,4], which boasts numerous advantages [5] and is valued for its high oil yield [6]. It also serves as a valuable timber and economic forest tree [7], and even the oil residue demonstrates remarkable performance in high-efficiency energy storage [8]. Consequently, *Idesia polycarpa* has garnered considerable attention from researchers across various fields [9,10]. The fruit of *Idesia polycarpa* is colorful and can beautify the environment, and its oil content can be as high as 43.6 % [11]. The oil extracted from the fruit is rich in unsaturated fatty acids, of which the content of linoleic acid can reach 75 %. *Idesia polycarpa* oil is semi-dry oil, which can be used as a high-quality edible oil [12,13].

Unsaturated fatty acids are prone to oxidation, hydrolysis and other reactions, resulting in hydroperoxides decomposed into ketones, aldehydes, acids and other substances, which not only affects the taste of the oil, but also harms human health. The commonly used method to slow down the oxidation rate of fats and oils is to add antioxidants. Guo et al. (2023) studied two synthetic antioxidants butylated hydroxytoluene (BHT) and propyl gallate (PG) added in *Idesia polycarpa* oil and found that PG exhibited good antioxidant effect in the oil [14]. Although there are many kinds of antioxidants and synthetic antioxidants are effective, they have a greater risk in terms of food safety, and their use is currently restricted. Therefore, natural antioxidants of plant origin are the optimal choice, and their core components not only possess antioxidant effects, but also have anti-inflammatory and bactericidal effects in some components [15]. Rosemary extract exhibits anti-oxidative effects by scavenging free radicals, while ascorbic palmitate can reduce the oxygen content in the oxidation system [16]. Tea polyphenol palmitate and Vitamin E has good anti-oxidative activity in *Idesia polycarpa* Maxim. oil [14,17,18].

However, most of the current studies on *Idesia polycarpa* oil have focused on the areas of extraction methods [5,19,20], oil quality analysis [21,22], and refining process [15,19,23], and few studies have been reported on its rancidity. It is unclear whether plant-derived antioxidants can slow oxidation in *Idesia polycarpa* oil and whether it is suitable to use under frying temperature (160 °C). Thus, this study conducted a comparative analysis of antioxidant effects of seven distinct natural antioxidants to identify the most effective one in *Idesia polycarpa* oil. Using response surface method, experiments were designed to compound the single antioxidants with good effects, obtaining the compound formula with best antioxidant effect, which was then verified under simulated frying temperature (160 °C). In the study, we aim to find out the most effective natural antioxidants to slow down the oxidation rate of *Idesia polycarpa* Maxim. oil and prove its effectiveness under frying temperature, which will improve the oxidative stability of *Idesia polycarpa* Maxim. oil, provide a theoretical basis for replacing synthetic antioxidants with technical guidance for antioxidant research, enhance the development and utilization of *Idesia polycarpa* Maxim. oil and broaden its application areas.

2. Materials and methods

2.1. Test materials and instruments

Fresh fruits of *Idesia polycarpa* Maxim. were purchased from Sichuan Youda *Idesia polycarpa* Maxim. Seed Co., Ltd.

Seven different natural antioxidants were applied in the experiment, all of which were of analytical grade and purchased from Shaanxi Haiyis Biotechnology Co., Ltd. (Xi'an, Shaanxi, China). Rosemary Extract (purity≥20 %); Tea Polyphenol Palmitate (purity≥98 %); Vitamin E (purity≥50 %); Ascorbyl Palmitate (purity≥98 %); Licorice Extract (purity≥30 %); Bamboo Leaf Extract (purity≥10 %); Black Wolfberry Extract (purity≥20 %).

Instruments used in the test:

Electronic Analytical Balance FA2004, Shanghai Shangping Instrument Co., Ltd;

High-Speed Refrigerated Centrifuge 3K18, Beijing Ocean Oriental Technology Development Co., Ltd.;

Blower-Type Constant Temperature Drying Oven (10 °C–300 °C), Shanghai Guangdi Instrument Equipment Co., Ltd.;

Temperature-Controlled Magnetic Stirrer XMTD-702, Jiangsu Jinyi Instrument Technology Co., Ltd.;

Dual-Temperature Home Oil Press WMTL-RZSA, Zhongshan Weimei Tianli Electric Appliance Co., Ltd.;

752 UV–Visible Spectrophotometer, Shanghai Jinghua Scientific Instrument Co., Ltd.

2.2. Test methods

2.2.1. Preparation of Idesia polycarpa oil

The fresh fruit of *Idesia polycarpa* were picked from the branches, removing bad and mouldy fruit. The picked fresh fruits were rinsed three times with clean water, wiped off the moisture and placed indoor to let it dry naturally. 100 pounds of *Idesia polycarpa* fruit were pressed using cold pressing mode, yielding 20 pounds of gross oil. The gross oil was centrifuged in a centrifuge at 10,000 r/min

Table 1
The maximum amount of antioxidants added.

Name of antioxidant	Additive Quantity(g/kg)
Rosemary Extract	0.7
Tea Polyphenol Palmitate	0.6
Vitamin E	0.4
Ascorbyl Palmitate	0.2
Licorice Extract	0.4
Bamboo Leaf Extract	0.4
Black Wolfberry Extract	0.4

for 15 min to separate the impurities from the oil and fat, and ultimately to obtain the upper layer of *Idesia polycarpa* oil free from impurities.

2.2.2. Design of the experimental programme

(1) Antioxidant effect of maximum addition of single antioxidant

According to GB 2760-2014 ″National Standard for Food Safety Standard for the Use of Food Additives" [24], the maximum dose of different antioxidants allowed to be added were added to 30 g of *Idesia polycarpa* oil (The amount of antioxidants added is shown in Table 1. For those without a specified maximum addition level in the National Standard, 0.4 g/kg was added.), and in order to enable the antioxidants to be fully dissolved in *Idesia polycarpa* oil, each sample was heated in 70 ± 5 °C waters for 5 min. The *Idesia polycarpa* oil added with antioxidant was placed in an oven at 60 ± 3 °C for Schaal accelerated oxidation test, which was used to accelerate the natural oxidation process and evaluate the oxidative stability of oils and fats. *Idesia polycarpa* oil without antioxidant was used as a control, and the samples were taken from the oven every 2 days (After removal, the samples were placed at room temperature). Samples were taken consecutively for 12 days, and their peroxide values were tested three times each time, respectively, and the average value was taken.

(2) Antioxidant effects of compounded antioxidants

Response surface method was used to design the experiments for the antioxidants to be compounded, using Design Expert software (8.0.6.1). D-Optimal was used to combine the antioxidants to be compounded, and the combination was designed in accordance with the national standard GB 2760-2014 “National Standard for Food Safety Standard for the Use of Food Additives”, which states that “when food additives with the same function are used in a mixture, the sum of the proportions of the respective dosages to the maximum dosage shall not exceed 1.” for design [24].

Natural antioxidants with a significant difference ($p < 0.05$) in peroxide value with the control group at all sampling points in the first single-factor experiment above were selected as test factors in the second experiment in this step. According to the results from the first experiment, rosemary extract (X1), tea polyphenol palmitate (X2), and ascorbyl palmitate (X3) were used as the three test factors, and the peroxide value (Y) was used as the response value, and the design yielded the levels of the test factors in Table 2.

The design of the experimental factors led to the design of 20 experimental combinations using D-Optimal (Table 3). In order to fully dissolve the antioxidants in *Idesia polycarpa* oil, each sample was heated at 70 ± 5 °C for 5 min.*Idesia polycarpa* oil with added antioxidants was placed in an oven at 60 ± 3 °C for Schaal accelerated oxidation test, and samples were taken after 7 days. The peroxide values were examined three times and the mean (Y) was taken.

(3) Antioxidant effects of compounding schemes at frying temperatures

The antioxidants of the compound formulation were added to *Idesia polycarpa* oil, while blank control was performed, and oven was used to simulate the temperature under frying (160 °C, [25–27]). The samples of *Idesia polycarpa* oil were placed in the oven and were taken every 6 h (After removal, the samples were placed at room temperature) for a total duration of 36 h. The samples were analyzed for peroxide value, acid value, anisidine value, malondialdehyde content and fatty acid composition.

2.2.3. Physicochemical and quality assessment of Idesia polycarpa oil

The assessment of peroxide value was measured by titration method with reference to GB 5009.227-2023 “National Food Safety Standard - Determination of Peroxide Value in Foods” [28]. The acid value was determined with reference to GB 5009.229-2016 “National Food Safety Standard - Determination of Acid Value in Foods” [29]. The anisidine value was determined with reference to GB/T 24304-2009 “Animal and Vegetable Fats and Oils-Determination of Anisidine Value” [30]. The assessment of Malondialdehyde was referred to the spectrophotometer method in the National Standard GB 5009.181-2016, “National Food Safety Standard - Determination of Malondialdehyde in Foods” [31]. The assessment of fatty acids was referred to GB 2716-2018 “National Food Safety - Edible Vegetable Oil” [32].

Table 2
Design of experimental factors.

Factor	Notation	Level of factors	
		−1	1
rosemary extract	X_1	0.00	0.70
tea polyphenol palmitate	X_2	0.00	0.60
ascorbyl palmitate	X_3	0.00	0.20

Note: Three-factor qualification: $0 \leq 10/7X_1+5/3X_2+5X_3 \leq 1$.

Table 3
Optimal scheme design table.

No.	X_1	X_2	X_3
1	0.35	0.00	0.10
2	0.35	0.30	0.00
3	0.00	0.20	0.13
4	0.00	0.00	0.10
5	0.35	0.00	0.1
6	0.00	0.00	0.00
7	0.47	0.00	0.07
8	0.35	0.00	0.00
9	0.00	0.30	0.00
10	0.17	0.15	0.05
11	0.00	0.00	0.20
12	0.35	0.00	0.00
13	0.35	0.30	0.00
14	0.00	0.30	0.00
15	0.00	0.00	0.10
16	0.23	0.20	0.00
17	0.00	0.20	0.07
18	0.70	0.00	0.00
19	0.00	0.30	0.10
20	0.00	0.60	0.00

3. Results

3.1. Comparative analysis of antioxidant effects of single antioxidant

In order to screen the natural antioxidants of *Idesia polycarpa* oil, the study was carried out to analyze the peroxide value of *Idesia polycarpa* oil spiked with seven different natural antioxidants dynamically for 288 h using Schaal's oven method. The analysis confirmed that the peroxide value of *Idesia polycarpa* oil both in the control group (no antioxidant added) and in different treatment groups showed an increasing trend with the extension of the treatment time (Fig. 1). Among them, *Idesia polycarpa* oil with the addition of rosemary extract, tea polyphenol palmitate, and ascorbyl palmitate showed a significant difference ($p < 0.05$) in peroxide value with the control at all sampling points. The addition of vitamin E and liquorice extracts showed significant difference in peroxide values with control at some of the treatment time points.The addition of bamboo leaf extract and black wolfberry extract had no significant effect on reducing the peroxide value of *Idesia polycarpa* oil. The results showed that single component rosemary extract, tea polyphenol palmitate and ascorbyl palmitate had significant effects on enhancing the antioxidant properties of *Idesia polycarpa* oil, with rosemary extract being the most effective (Fig. 1, Table S1)

3.2. Antioxidant effects of compound antioxidants

In order to analyze the antioxidant effect of the compound antioxidants, the corresponding peroxide values were determined for

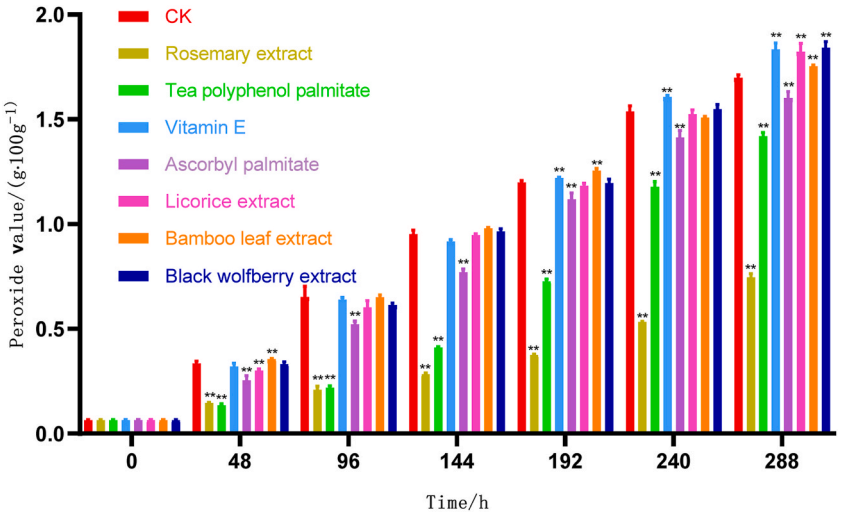

Fig. 1. Effects of different antioxidants on peroxide value of *Idesia polycarpa*oil *significant at $p < 0.05$ level.

each of the 20 different formulated test combinations of *Idesia polycarpa* oil treated for 7 days as devised by D-Optimal, and the mean (Y) of the three measurements was taken separately. The experimental results were analyzed by multiple regression using Design Expert software (8.0.6.1) to establish a quadratic response model. Based on the analysis of significance in Table 4, a quadratic multiple regression equation was obtained with peroxide value as the response value (Y):

$Y = 0.89737–0.55868X1-0.56826X2-1.97266X3+0.42649 \times 1 \times 2+3.02384 \times 1 \times 3+4.15224 \times 2 \times 3+0.34777X12 + 0.46719X22 + 6.02858X32$. The model difference was highly significant ($p < 0.0001$), the misfit term was not significant ($p > 0.05$), and the fit R^2 was close to 1, which proved that the model was well fitted and the regression equation could be used to analyze the effect of the factors on the peroxide value.

The selection of the optimal solution was carried out by Design Expert software (8.0.6.1) to minimize the Y value of the equation, and the best antioxidant formulation was screened as a complex of rosemary extract with tea polyphenol palmitate. The optimized formulation of the complex antioxidant was obtained as 0.45 g/kg of rosemary extract addition with 0.21 g/kg of tea polyphenol palmitate addition, and the peroxide value predicted by the equation was 0.6564 g/100g. The best compound formulation obtained was validated by performing three repetitive operational experiments, and the peroxide value of *Idesia polycarpa* oil obtained under this formulation was in general agreement with the predictions derived from the model, which proved the feasibility of the formulation.

3.3. Effect of addition of compound antioxidants on the quality of Idesia polycarpa oil under frying temperature (160 °C)

In order to analyze the high-temperature stability of the compound antioxidant-added *Idesia polycarpa* oil, the study was carried out to determine the dynamic trends of the peroxide value, acid value, anisidine value, Malondialdehyde content and fatty acid composition of the compound antioxidant-added *Idesia polycarpa* oil (T) under the condition of 160 °C, respectively, and to make a comparison with that of the non-antioxidant-added *Idesia polycarpa* oil (CK). The results confirmed that: during heating, (1) The peroxide values of the antioxidant-added group were lower than those of the non-added group, with significant differences at 6h, 18h, 30h and 36h (Fig. 2A, Table S2); (2) Both groups showed a fluctuating change in acid values with a decrease followed by an increase, but the difference was not significant (Fig. 2B–Table S2); (3) Anisidine values showed an increasing trend in both groups. The increase was faster in the first 6h, after which the increase slowed down. Anisidine values were lower in the antioxidant-added group than in the non-added group at all measured points and were significantly different (Fig. 2C–Table S2); (4) The Malondialdehyde content of both groups showed an increasing trend, the Malondialdehyde content of the non-added group increased faster in the first 6h and after the 30h, and the Malondialdehyde content of the added group was basically flat and no longer increased after 18h. The Malondialdehyde content of the added group was lower than that of the non-added group at all measured points, with significant differences except at 18h (Fig. 2D–Table S2). This suggests that the addition of compound antioxidants to *Idesia polycarpa* oil inhibited the formation of Malondialdehyde; (5) With the increase of heating time, the proportions of hexanoic acid, decanoic acid, myristic acid, palmitoleic acid, and stearic acids remained more or less the same in both groups, the proportions of octoic acid, palmitic acid, and oleic acids increased, and the proportions of linoleic acid and linolenic acid decreased. At 36h, the proportion of linoleic and linolenic acids was higher in the added group than in the non-added group, and dodecaenoic acid was not detected in the added group. It can be seen that the rate of unsaturated fatty acid loss was slower in the added group than in the non-added group, but the difference was not significant (Table 5).

(A: the dynamic changes of peroxide value; B: the dynamic changes of acid value; C:the dynamic changes of Malonaldehyde content; D:the dynamic changes of fatty acid; T: *Idesia polycarpa* oil with compound antioxidant; CK: *Idesia polycarpa* oil without

Table 4
Variance analysis of antioxidant stability of *Idesia polycarpa* oil.

Source of variance	Sum of squares	Degree of freedom	Mean square	F value	Prob > F
Model	0.049	9	5.42 × 10-3	54.78	<0.0001**
X_1	2.88 × 10-4	1	2.88 × 10-4	2.91	0.1186*
X_2	1.15 × 10-3	1	1.15 × 10-3	11.61	0.0067**
X_3	4.14 × 10-3	1	4.14 × 10-3	41.83	<0.0001**
X_1X_2	8.61 × 10-4	1	8.61 × 10-4	8.7	0.0145*
X_1X_3	5.13 × 10-3	1	5.13 × 10-3	51.8	<0.0001**
X_2X_3	6.41 × 10-3	1	6.41 × 10-3	64.8	<0.0001**
X_1^2	2.08 × 10-3	1	2.08 × 10-3	20.98	0.001**
X_2^2	2.12 × 10-3	1	2.12 × 10-3	21.44	0.0009**
X_3^2	4.20 × 10-3	1	4.20 × 10-3	42.49	<0.0001**
Residual	9.89 × 10-4	10	9.89 × 10-5		
Misfit term	7.33 × 10-4	5	1.47 × 10-4	2.86	0.1369
Absolute Deviation	2.57 × 10-4	5	5.13 × 10-5		
Sum	0.05	19			
Statistical parameters of the model					
Standard deviation	9.95 × 10-3		Degree of fitting R^2	0.9801	
Mean	0.74		Correction of fit R^2	0.9622	
Correction value(%)	1.35		Prediction R^2	0.7332	
Prediction error	0.013		Signal to Noise Ratio	33.636	

Note: * represents significant at the 0.05 level; ** represents highly significant at the 0.01 level.

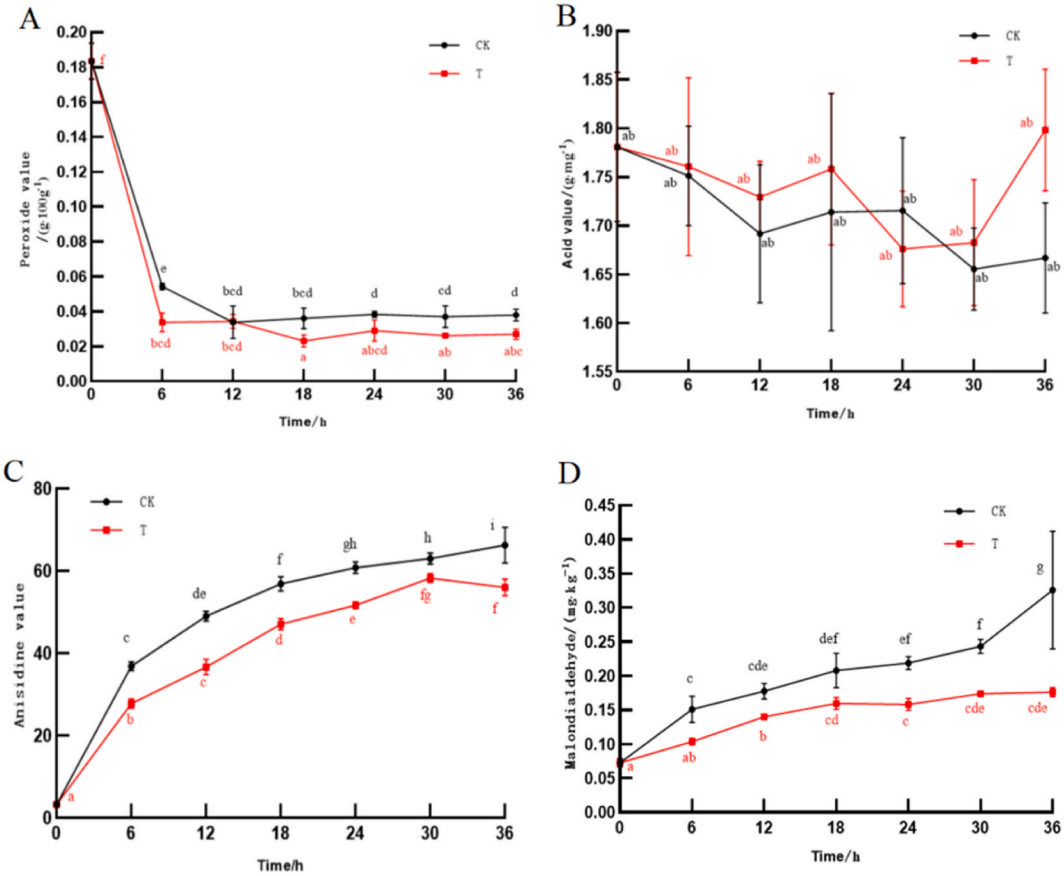

Fig. 2. Changes of peroxide value of *Idesia polycarpa* oil under different treatments at 160°C
Note: Lowercase letters in Fig. 2 indicate analysis of variance.

compound antioxidant)

4. Discussion

4.1. Effect of different antioxidants on Idesia polycarpa oil

By comparing seven different antioxidants, the best antioxidant was rosemary extract (Fig. 1, Carnosic acid>20 %). Carnosic acid is the main antioxidant component of rosemary extract [33], which can effectively scavenge free radicals to prevent the oxidation of *Idesia polycarpa* oil. The addition of vitamin E and black wolfberry extract to *Idesia polycarpa* oil did not have a significant antioxidant effect, and after 288 h of accelerated oxidation, vitamin E, liquorice extract, bamboo leaf extract and black wolfberry extract did not inhibit the oxidation of *Idesia polycarpa* oil but rather accelerated the oxidation (Fig. 1). Liu et al. [34] found that the addition of vitamin E to walnut oil had unsatisfactory antioxidant effects. Jung et al. [35] studied the addition of different concentrations of α-tocopherol to soybean oil and found that α-tocopherol can be antioxidant when the concentration is below 250 mg/kg, but it promotes the oxidation when the concentration is over 500 mg/kg. Whether vitamin E shows antioxidant effect is related to the fatty acid composition of lipids, but excessive addition of vitamin E can lead to its promotion of lipids oxidation [36]. *Idesia polycarpa* oil contains certain amount of vitamin E, which has certain self-antioxidant effect, so adding too much vitamin E will speed up its oxidation. Flavonoids in licorice and bamboo leaf extracts are their main antioxidant components [37], while the main antioxidant component in black wolfberry extract is anthocyanin, and the amount of the active ingredients in these three antioxidants may be not sufficient to provide effective antioxidant capacity. It is also possible that during the process of adding antioxidants, they are not completely dissolved in *Idesia polycarpa* oil, resulting in failure to provide desired antioxidant effect. In particular, anthocyanins are water-soluble pigments, and if they are to be used as antioxidants in lipids, they need to be modified to make them soluble in lipids. In addition, not only antioxidant components but also other components may be present in these extracts, which may play an accelerated oxidizing effect, thus leading to a greater peroxide value of *Idesia polycarpa* oil in the antioxidant-added group than the value in the control group at 288h (Fig. 1).

Table 5
Fatty acid composition of different treatments (%).

Fat Acid	Time/h													
	Non-added group							Added group						
	0	6	12	18	24	30	36	0	6	12	18	24	30	36
hexanoic acid	0.03	0.02	0.03	0.04	0.03	0.04	0.04	0.03	0.03	0.03	0.04	0.03	0.03	0.03
octoic acid	–	0.02	0.03	0.04	0.04	0.05	0.05	–	0.02	0.03	0.03	0.03	0.04	0.04
decanoic acid	0.07	0.05	0.06	0.07	0.05	–	0.07	0.07	0.01	0.07	0.09	0.05	0.06	0.07
myristic acid	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07
palmitic acid	12.59	12.6	12.76	12.83	12.82	13.00	13.00	12.59	12.64	12.69	12.82	12.8	12.89	12.90
palmitoleic acid	2.43	2.42	2.42	2.43	2.43	2.48	2.46	2.43	2.41	2.43	2.43	2.44	2.45	2.45
stearic acid	1.14	1.15	1.15	1.16	1.16	1.20	1.18	1.14	1.15	1.15	1.17	1.17	1.17	1.17
oleic acid	10.41	10.55	10.50	10.80	10.83	10.89	10.85	10.41	10.63	10.65	10.80	10.70	10.79	10.82
Linoleic acid	72.21	72.05	71.94	71.70	71.61	71.41	71.33	72.21	72.00	71.86	71.68	71.84	71.50	71.59
linolenic acids	0.92	0.90	0.88	0.87	0.85	0.85	0.84	0.92	0.90	0.89	0.87	0.87	0.86	0.86
dodecaenoic acid	0.12	0.17	0.15	–	0.11	–	0.12	0.12	0.15	0.12	–	–	0.13	–
saturated fatty acid	13.9	13.91	14.1	14.21	14.17	14.36	14.41	13.9	13.92	14.04	14.22	14.15	14.26	14.28
unsaturated fatty acid	86.09	86.09	85.89	85.8	85.83	85.63	85.6	86.09	86.09	85.95	85.78	85.85	85.73	85.72

Note: "-" indicates that the substance was not detected.

4.2. Response surface methodology analysis

Based on the results from the single-factor experiment in 2.2.2 (1), three natural antioxidants, which were significantly lower ($p < 0.05$) in peroxide value than the control group at all sampling points, were selected as test factors (X1: rosemary extract, X2: tea polyphenol palmitate, X3: ascorbyl palmitate), and the corresponding peroxide value (Y) was used as the response value in the response surface methodology (RSM) optimization experiment. The design of the test factors led to the design of 20 experimental combinations using D-Optimal (Table 3). The experimental results were analyzed by multiple regression using Design Expert software (8.0.6.1) to establish a quadratic response model. Based on the analysis of significance in Table 4, a quadratic multiple regression equation was obtained:

$Y = 0.89737–0.55868X1-0.56826X2-1.97266X3+0.42649 \times 1 \times 2+3.02384 \times 1 \times 3+4.15224 \times 2 \times 3+0.34777X1^2+0.46719X2^2+6.02858X3^2$. From the analysis of variance (Table 4), it could be seen that the model difference was highly significant ($p < 0.0001$), indicating that the model reached a highly significant level. The misfit term was insignificant ($p = 0.1369 > 0.05$), indicating that the model had a good fit. Also, the fit R^2 was close to 1, which proved that the model was well fitted and the regression equation could be used to analyze the effect of the factors on the peroxide value.

The selection of the optimal solution was carried out by Design Expert software (8.0.6.1) to minimize the Y value of the equation, and the best antioxidant formulation was screened as a complex of rosemary extract and tea polyphenol palmitate. The optimized formulation of the complex antioxidant was obtained as 0.45 g/kg of rosemary extract addition with 0.21 g/kg of tea polyphenol palmitate addition, and the peroxide value predicted by the equation was 0.6564 g/100g. The best compound formulation obtained was validated by performing three repetitive operational experiments, and the peroxide value of *Idesia polycarpa* oil obtained under this formulation was in general agreement with the predictions derived from the model, which proved the feasibility of the formulation.

4.3. Effect of heating at 160 °C on Idesia polycarpa oil

Lipids undergo reactions such as oxidation and hydrolysis of unsaturated fatty acids during frying. The peroxide value of *Idesia polycarpa* oil supplemented with compounded antioxidants decreased sharply at 160 °C and then flattened out after slight upward and downward fluctuations. Wang [38] analyzed the changes in the peroxide value of camellia seed oil during heating at high temperatures, and the peroxide value of camellia seed oil with the addition of rosemary extract first increased and then gradually decreased during the heating process, which is contrary to the results of this study. The reason for such a result may be due to the fact that some peroxides are produced in the pressing process of *Idesia polycarpa* oil, and these peroxides accelerate the decomposition in the process of high temperature heating, and the speed of producing new peroxides is lower than the decomposition speed, and when the speed of producing free radicals in the oil is the same as the speed of consuming the free radicals, the peroxide value of *Idesia polycarpa* oil tends to be stabilized gradually.

Idesia polycarpa oil showed an up and down trend in acid value during high temperature heating, which is in accordance with the results of Mu [39] on high temperature heating of frying oil. Lipids undergo complex chemical changes during heating, and the decomposition of hydroperoxide increases free fatty acids, but at the same time small molecules of fatty acids are volatilized, resulting in a decrease in acid value. However, after 36 h of high-temperature heating, the acid value of *Idesia polycarpa* oil with added compound antioxidants was lower than the requirement of GB 2716-2018 “National Food Safety-Edible vegetable oil” [32] for the acid value of edible oils during frying (5 mg/g).

Guo [40] examined anisidine in tea seed oil during frying and concluded that the anisidine value increased with frying time. The results of the this study are in agreement with the above that heating at high temperature causes a sharp increase in anisidine value. The anisidine value mainly represents the amount of aldehydes and ketones produced in lipids. As the heating time increases, the fatty acids decompose to produce mainly α- and β-unsaturated aldehydes, and the anisidine value increases. However, aldehydes also decompose gradually under high temperature conditions [41], and the increase in anisodamine value slows down after heating up to 30 h. Due to the addition of antioxidants, aldehydes are generated at a slower rate, and there is a tendency for anisodamine value to decrease.

Malondialdehyde is a secondary product of high-temperature heating of lipids, and the rancid odour produced after the rancidity of lipids mainly refers to malondialdehyde. The larger the ratio, the stronger the irritating odour produced by the rancidity. The consumption of oils with high content of malondialdehyde will have a great impact on the human body, and malondialdehyde can be accumulated in the body and disrupted the original redox balance in the body, resulting in an imbalance in the organism and triggering diseases [42–44]. The addition of compound antioxidants can effectively slow down the production of malondialdehyde and improve the quality of *Idesia polycarpa* oil as frying oil.

With the increasing time of heating *Idesia polycarpa* oilat high temperatures, the unsaturated fatty acids, especially linoleic acid, were oxidized and decomposed into valeraldehyde, hexanal, heptaldehyde, pentanol, hexanoic acid, octanoic acid, methyl ketone, and ketenes [45], which led to a decrease in the proportion of linoleic acid. Saturated fatty acids are also oxidized to produce aldehydes and ketones at temperatures above 150 °C [46], but since the main fatty acid in *Idesia polycarpa* oil is linoleic acid, the proportion of unsaturated fatty acids decreases and the proportion of saturated fatty acids increases. Wei [47] found that linoleic and linolenic acid content decreased after peony seed oil was subjected to high-temperature heating; Zhou [48] heated grape seed oil and walnut oil at high temperature with 160 °C, the content of linoleic acid and linolenic acid decreased, and the content of oleic acid and palmitic acid increased; Cheng [49] found that in heating studies on linseed oil, walnut oil, and avocado oil, their fatty acid content the unsaturated fatty acid content of linseed oil, walnut oil, and avocado oil decreased and the saturated fatty acid content increased with the increase

of the heating time at 200 °C. The changes of fatty acid upon heating of *Idesia polycarpa* oil in this study were consistent with the above results.

5. Conclusions

Idesia polycarpa oil is high in linoleic acid and is a high quality edible vegetable oil. In this study, the antioxidant effects of seven different natural antioxidants on *Idesia polycarpa* oilwere compared experimentally, and it was concluded that rosemary extract was the natural single antioxidant with more pronounced antioxidant effects. Through the optimization test on the addition amount of three different antioxidants, rosemary extract, tea polyphenol palmitate and ascorbyl palmitate, the best compound formulation was obtained as 0.45 g/kg rosemary extract +0.21 g/kg tea polyphenol palmitate, which can effectively retard the oxidation rate of *Idesia polycarpa* oil. Meanwhile, the antioxidant capacity of *Idesia polycarpa* oil added with compound antioxidants was significantly improved under high temperature conditions, and the generation of secondary oxidation products was inhibited and the loss of unsaturated fatty acids was reduced. Therefore, *Idesia polycarpa* oil with added compound antioxidants can be used as frying oil, which provides a theoretical basis for the storage and consumption safety of *Idesia polycarpa* oil and improves the safety of *Idesia polycarpa* oil as edible oil. The development and application of the natural compound formula will contribute to the establishment of quality standards for *Idesia polycarpa* oil as edible oil. Further study on the shelf life of *Idesia polycarpa* oil could be conducted to enhance its storage stability.

CRediT authorship contribution statement

Fangming Liu: Writing – original draft, Visualization, Conceptualization. **Yurui Xie:** Data curation. **Xiaodong Geng:** Resources. **Zhi Li:** Methodology. **Yanmei Wang:** Project administration. **Zhen Liu:** Supervision. **Qifei Cai:** Writing – review & editing.

Data availability statement

Data will be made available on request.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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