Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

Fatty acid wax from epoxidation and hydrolysis treatments of waste cooking oil: Synthesis and properties

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List

Table S1. The optimization of the reaction temperature and reaction time in the hydrolysis process. Two times of the theoretical amount of NaOH was used for each sample (26.72 g NaOH for 100 g E-WCO).

Serial number	Temperature (℃)	Time (h)	Free fatty acid content (wt%)
A	50	4	68.08
B	60	4	80.29
C	70	4	96.41
D	80	4	96.47
E	90	4	96.21
F	70	1	55.94
G	70	2	68.04
H	70	3	83.29
I	70	5	96.58

The optimized hydrolysis temperature and time should be 70 °C and 4 h respectively, which could afford a high free fatty acid content of the wax (96.41 wt%, sample C, i.e. A6 sample). And is notable that, further increasing the reaction temperature and time (samples D and I) could not significantly improve the fatty acid content of the sample but might lead to increased safety risks and costs.

Table S2. Brief introduction of China Standards concerned in this paper.

Serial number		Brief introduction of the Standard
GB/T 5532-2008	Name	Animal and vegetable fats and oils - Determination of iodine value
	Principle	The sample is dissolved in a solvent and reacted with a Wechsler reagent. After a certain period of time, by adding potassium iodide and water, the precipitated iodine is titrated with sodium thiosulfate solution.
	Process	The sample is weighed into a 500 mL conical flask, and then an appropriate amount of solvent is added to dissolve the sample. Then, 25 mL of Weyers reagent is added accurately with a pipette. The conical flask is instantly sealed and shaken well and placed the conical flask in a dark place to react for 1 h. Except for not adding the sample, a blank solution is made according to the above steps. When reaching the prescribed reaction time, 20 mL potassium iodide solution and 150 mL deionized water is added. The titration process is using the calibrated sodium thiosulfate solution until the yellow color of the iodine is close to disappearing, and then, by adding a few drops of starch solution, the titration is continued by shaking the conical flask vigorously until the blue color just disappears. The iodine value of the sample is calculated according to equation (1): $$W_1 = \frac{12.69 \times c \times (V1 - V2)}{m} \quad (1)$$ W_1: the iodine value of the sample, expressed in grams of iodine per 100g of sample aspirated (g/100g); c: the concentration of a standard solution of sodium thiosulfate (mol/L); V_1: the volume of sodium thiosulfate standard solution consumed by the blank solution (mL); V_2: the volume of sodium thiosulfate standard solution consumed by the sample solution (mL); m: mass of the sample (g).

Serial number	Brief introduction of the Standard	
GB/T 1677-2008	Name	Determinating the epoxy value of plasticizers
	Principle	The epoxide value is the determination of the amount of oxygen in the ethylene oxide group in the specimen. After adding the hydrochloric acid-acetone solution to the specimen and reacting for a certain time, the remaining hydrochloric acid is titrated with sodium hydroxide-ethanol standard solution.

Serial number		Brief introduction of the Standard
	Process	0.5–1.0 g sample is precisely weighed (accurate to 0.0001g) and put in a 250ml conical flask with a stopper. And then, 20 mL of hydrochloric acid-acetone solution is added, The conical flask is instantly sealed and shaken well, and placed in a dark place for 30 min. Using phenolphthalein as an indicator, the solution is titrated with 0.15 mol/L NaOH-ethanol standard solution until pink. A blank experiment is carried out at the same time. The epoxy value is expressed in terms of oxygen content and is calculated according to equation (2): $$E = \frac{[V - (V_1 - \frac{V_2}{G} \times W)]N \times 1.6}{W}$$ (2) V: the volume of NaOH-ethanol titration solution consumed in the blank test (mL); V_1: the volume of NaOH-ethanol titration solution consumed in the sample test (mL); V_2: Volume of NaOH-ethanol titration solution consumed for acid value determination of the sample (mL); N: concentration of NaOH-ethanol titration solution (mol/L); W: mass of the sample (g); G: The mass of the sample in the determination of acid value (g).
GB/T 5534-2008	Name	Animal and vegetable fats and oils-Determination of saponification value
	Principle	The saponification value is a measure of the amount of free fatty acids and glycerides in oils and fatty acids. The sample is boiled under reflux conditions together with a sodium hydroxide-ethanol solution, in which the excess sodium hydroxide is then titrated with a hydrochloric acid standard solution.

Serial number		Brief introduction of the Standard
	Process	A certain mass of the sample is weighed into a conical flask, then 25 mL sodium hydroxide-ethanol solution was added precisely, along with some zeolite. This mixture is heated to reflux for 1 h. After the reaction is completed, 0.5–1 mL phenolphthalein indicator is added to the hot solution, and then the solution is titrated with the hydrochloric acid standard solution until the indicator's pink color just disappears. A blank experiment is carried out at the same time. The saponification value of the sample is calculated according to Equation (3): $I = \frac{(V_0 - V_1) \times c \times 40}{m}$ (3) *I*: saponification value (in NaOH) (g/g) V_0: volume of a standard solution of hydrochloric acid consumed in the blank experiment (mL) V_1: volume of a standard solution of hydrochloric acid consumed by the sample (mL) *c*: concentration of a hydrochloric acid standard solution (mol/L) *m*: mass of the sample (g)
GB/T 14571.3-2008	Name	Ethylene glycol for industrial use - Determination of content of total aldehydes present - Spectrophotometric method
	Principle	In the presence of ferric chloride, the aliphatic aldehyde in the sample reacts with 2-methyl-2-benzothiazolone hydrazone (MBTH) to produce a blue-green thickened cation, and the absorbance is measured by spectrophotometer at a wavelength of 620 nm.

Serial number		Brief introduction of the Standard
	Process	At first, a working curve should be plotted: In six 50 mL volumetric flasks, 0 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, and 5.0 mL standard solution are added, and then 5.0 mL, 4.0 mL, 3.0 mL, 2.0 mL, 1.0 mL, and 0 mL water are added and shook well, respectively. Then 5.0 mL of MBTH solution is added to each solution and shaken well, and the reaction was carried out at room temperature for 30 min. 5.0 mL of oxidant solution (mixture of 1% ferric chloride and 1.2% sulfamic acid) is added to each solution and reacted for 20 min. Finally, the absorbance was diluted to the scale with distilled water and measured at 620 nm with water as the reference solution. The working curve was plotted with the mass of formaldehyde (μg) as the horizontal coordinate and the corresponding net absorbance (deducting the absorbance of the reagent blank) as the vertical coordinate. The equation of the working curve is expressed as $C=K\times X\times A+B$, and the correlation coefficient should be greater than 0.99. Then, an appropriate amount of sample is weighed (accurate to 0.0002 g) and dissolved in a 50 mL volumetric flask and tested following the above steps. A blank experiment is carried out at the same time. On the working curve equation, the mass of aldehyde (μg) was calculated from the net absorbance, and then the mass of aldehyde (in formaldehyde) in the specimen was calculated according to equation (4). $w = \frac{m_1}{m}$ (4) w: mass of aldehyde per 1g sample (μg/g) m_1: mass of aldehyde found on the working curve (μg) m_2: mass of the sample (g)
GB/T 5009.168-2016	Name	National Food Safety Standard - Determination of fatty acids in foods

Serial number	Brief introduction of the Standard
	Principle: Ester exchange method: the oil and grease sample is dissolved in isooctane, and then potassium hydroxide-methanol solution is added for a methylesterification reaction. when the reaction is complete, sodium bisulfate is used to neutralize the remaining potassium hydroxide. The percent fatty acid content is determined by the area normalization method.
	Process: The sample is dissolved in 4 mL isooctane, and then 200 μL of potassium hydroxide-methanol solution is added. Cover with a glass stopper, the mixture is shaken vigorously for 30 s, and let stand until clarified. Then 1 g of sodium bisulfate is added shaking vigorously to neutralize the potassium hydroxide. After the salt precipitates, the upper layer of the solution is transferred to the upper machine bottle and waited for measurement. The percentage Y_i of a particular fatty acid in the sample to the total fatty acid is calculated according to equation (5), and the content of a given component *i* is calculated by determining the percentage of the corresponding peak area to the sum of the peak areas of all components. $$Y_i = \frac{A_{Si} \times F_{FAME_i - FA_i}}{\sum A_{Si} \times F_{FAME_i - FA_i}}$$ (5) Y_i: the percentage (%) of a fatty acid of the total fatty acids in the specimen. A_{si}: the peak area of each fatty acid methyl ester in the specimen determination solution. $F_{FAME_i - FA_i}$: the coefficient of conversion of fatty acid methyl ester i into fatty acid. $\sum A_{Si}$: the sum of the peak areas of each fatty acid methyl ester in the sample determination solution.

Serial number		Brief introduction of the Standard
GB/T 30392-2013	Name	Vegetable wax
	Principle	The performance of vegetable wax must meet the following indicators: 1. Smell: no peculiar smell. 2. Lovibond color codes: Y ≤ 35 and R ≤ 4. 3. Water and volatile matter concentration: ≤ 0.5% 4. Melting point: Plant wax can be divided into four types, i.e. type I (40 ℃–50 ℃), type II (51 ℃–55 ℃), type III (56 ℃–60 ℃) and type IV (61 ℃–70 ℃).
	Process	The Lovibond color codes of the plant vegetable can be tested by a Lovibond tintometer. The samples should be heated to melt all before testing and should be poured into the 133.4 mm colorimetric groove during the heat, and test the color. The whole colorimetric process should ensure that the sample does not solidify.

Table S3. The melting temperatures of WCO-based waxes and other control samples.

Sample	Initial melting Temperature (℃)	Complete melting temperature (℃)
WCO	33	36
E-WCO	36	40
A1	35	46
A2	39	50
A3	41	52
A4	43	54
A5	46	55
A6	44	53
SW	51	54
ref.44	46	49

Table S4. The Lovibond color codes of WCO-based waxes and other control samples.

Sample	Y (Yellow)	R (Red)	B (Blue)	Light field	Darkfield
WCO	52.3	24.8	2.2	0	0
E-WCO	34.5	10.7	0	0.6	0
A1	26.2	5.1	0	0	1
A2	25.1	4.9	0	0	0.8
A3	24.7	4.4	0	0	0.8
A4	18.9	3.7	0	0	0
A5	16.5	2.5	0	0	2
A6	11.9	2.3	0	0	1
SW	12.1	0.7	0	0	0
ref.44	16.1	2.3	0	0	0.6

Table S5. The penetration indexes of WCO-based waxes and other control samples.

Sample	Penetration index (mm)
WCO	-
E-WCO	23.15±0.27
A1	7.78±0.11
A2	5.16±0.12
A3	4.64±0.15
A4	3.78±0.14
A5	2.81±0.11
A6	2.66±0.16
SW	5.46±0.12
ref.44	2.95±0.15

Table S6. The combustion times of WCO-based waxes and other control samples.

Sample	Combustion time (min)
WCO	-
E-WCO	37±3
A1	163±5
A2	202±6
A3	223±8
A4	251±7
A5	288±5
A6	293±4
SW	276±9
ref.44	227±8

Table S7. The aldehydes contents of WCO-based wax and other control samples.

Sample	Aldehyde content (μg/g)
WCO	3.84
E-WCO	0.844
A6	7.98×10^{-2}
SW	3.84×10^{-2}

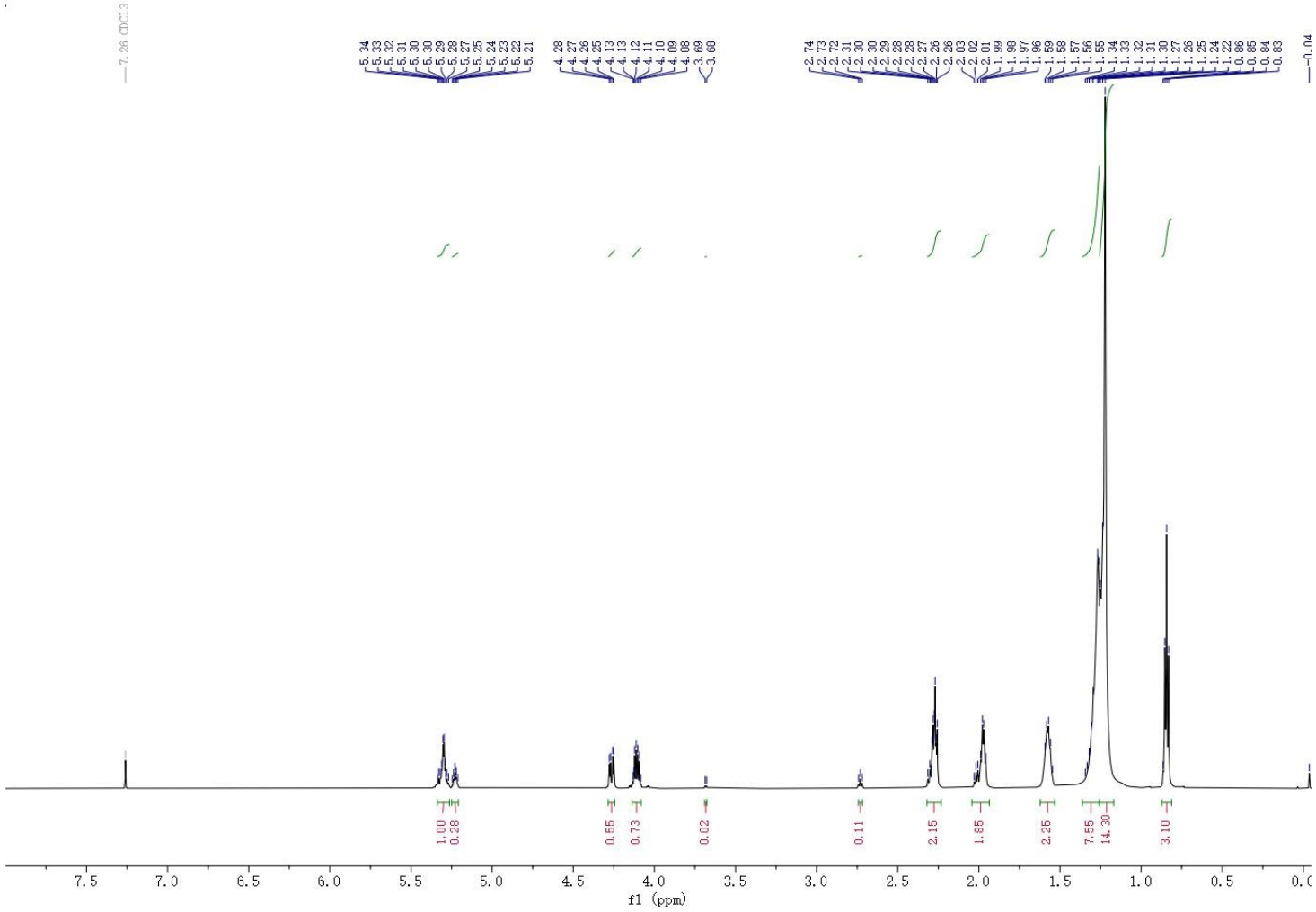

Figure S1. ^{1}H NMR spectra of WCO with integration and peak picking.

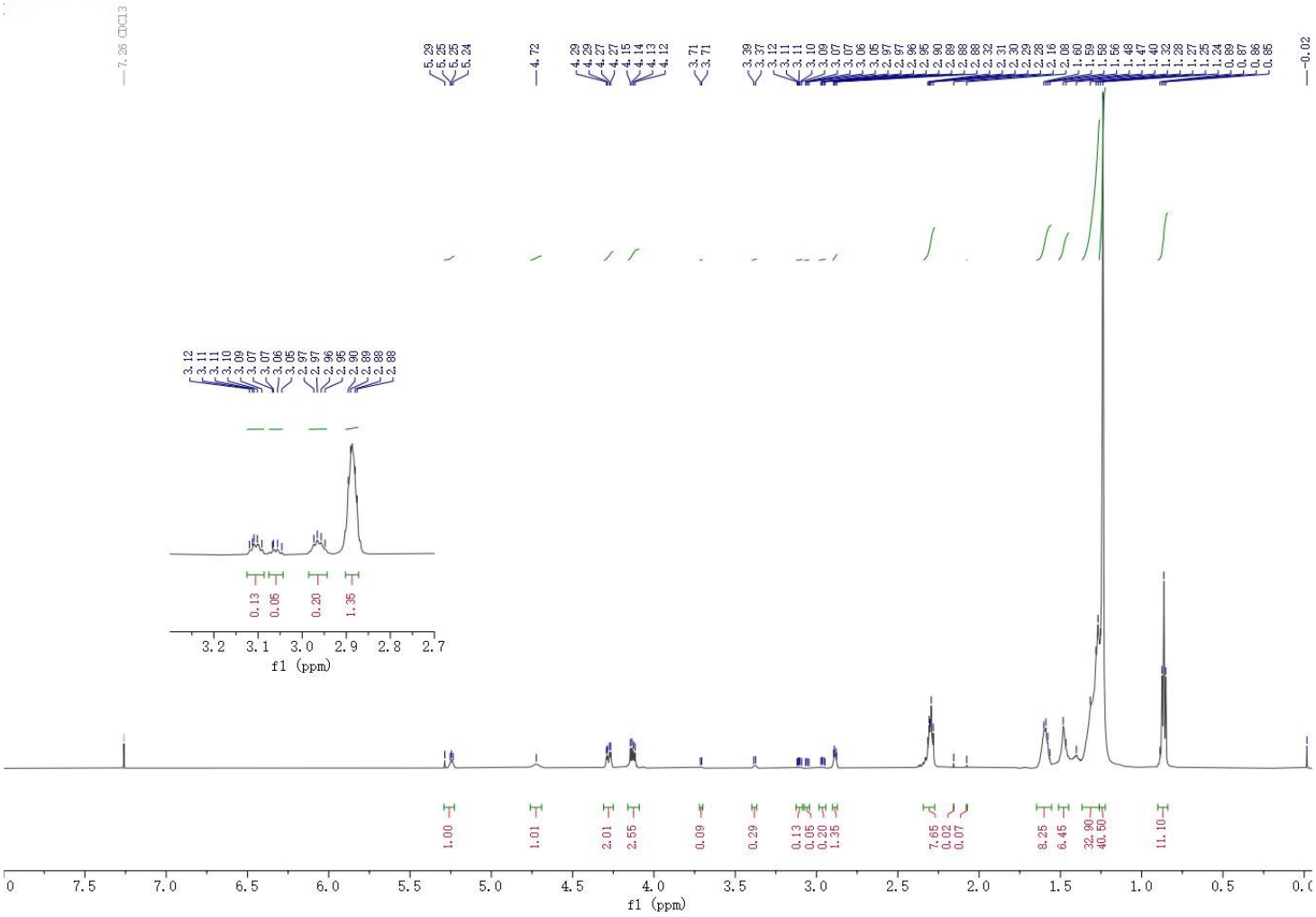

Figure S2.[1]H NMR spectra of E-WCO with integration and peak picking.

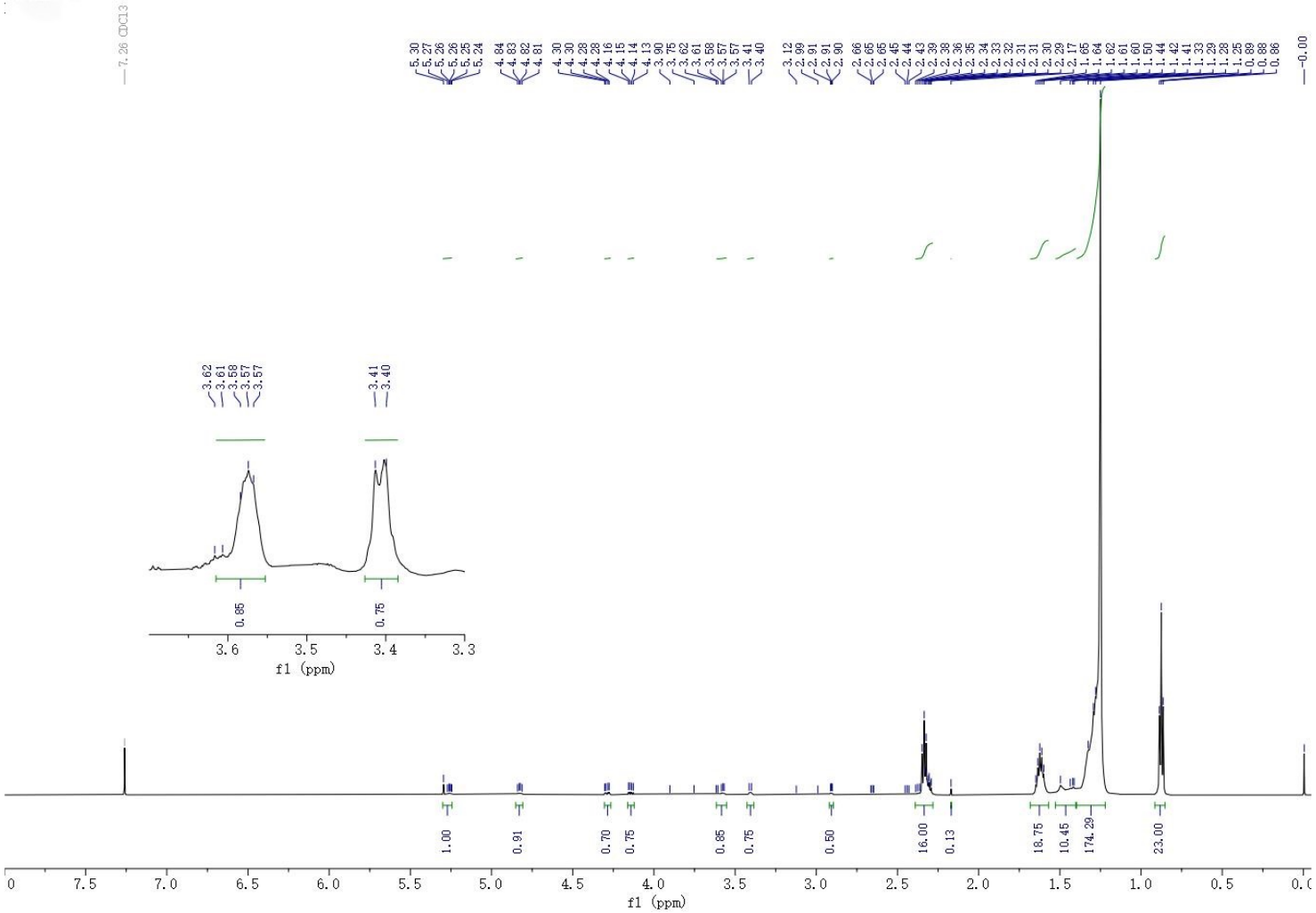

Figure S3. ^{1}H NMR spectra of A6 sample with integration and peak picking.

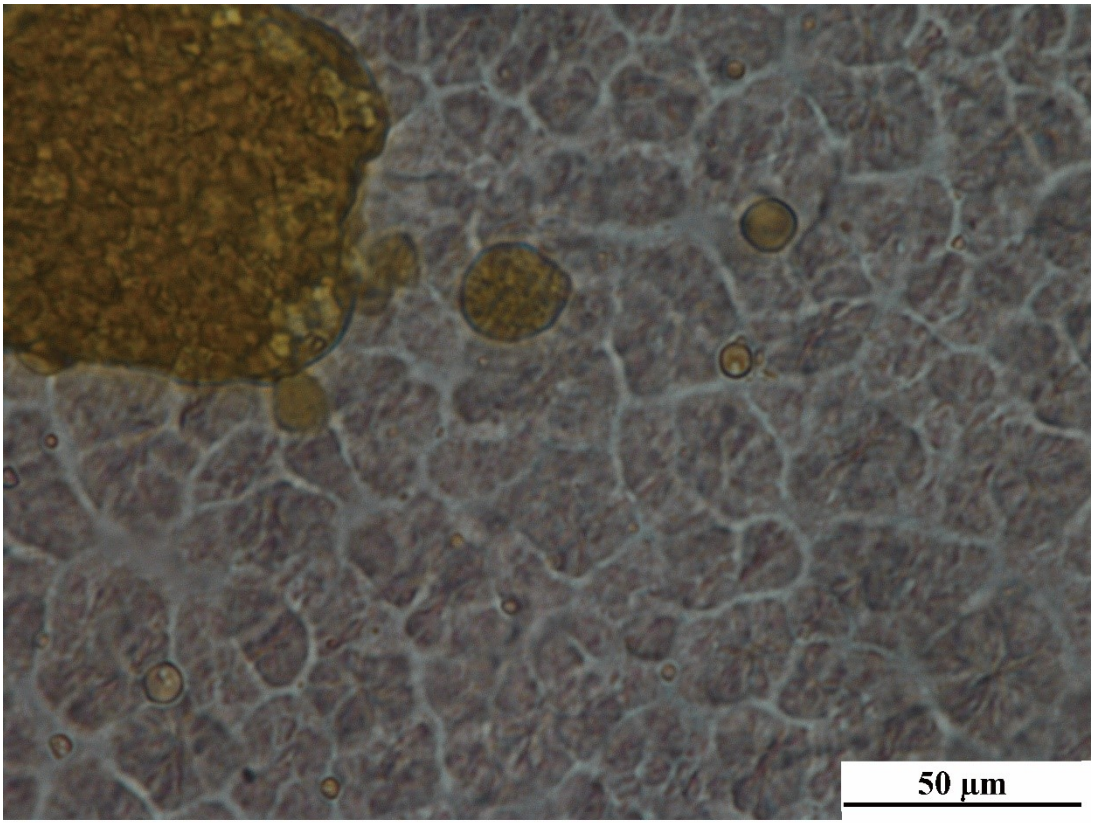

Figure S4. The 500× microphotograph of WCO.

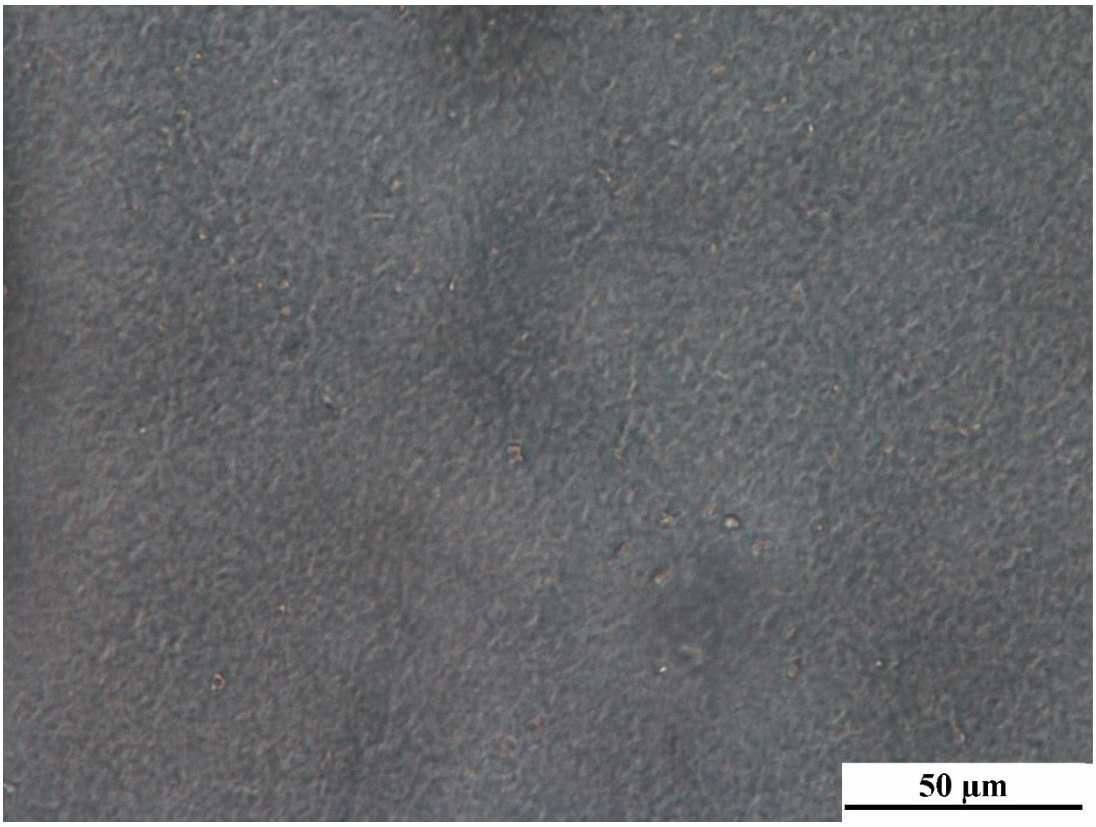

Figure S5. The 500× microphotograph of E-WCO.

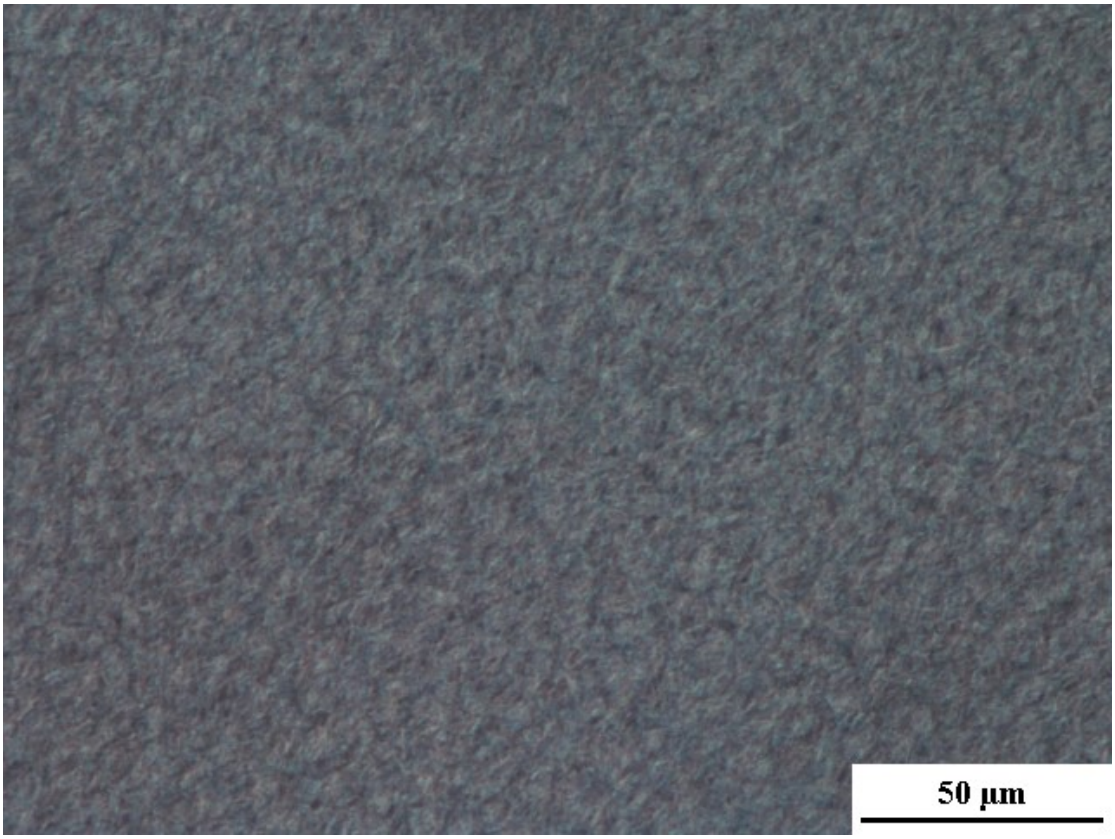

Figure S6. The 500× microphotograph of A1 sample.

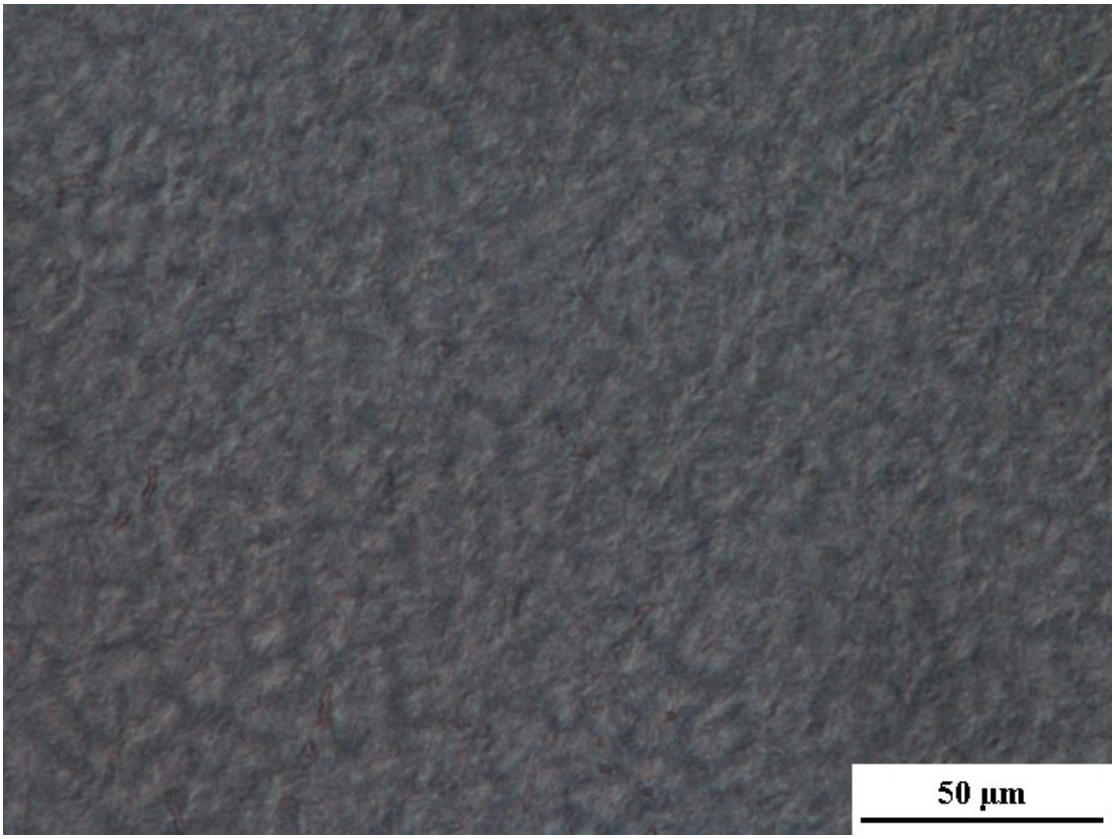

Figure S7. The 500× microphotograph of A2 sample.

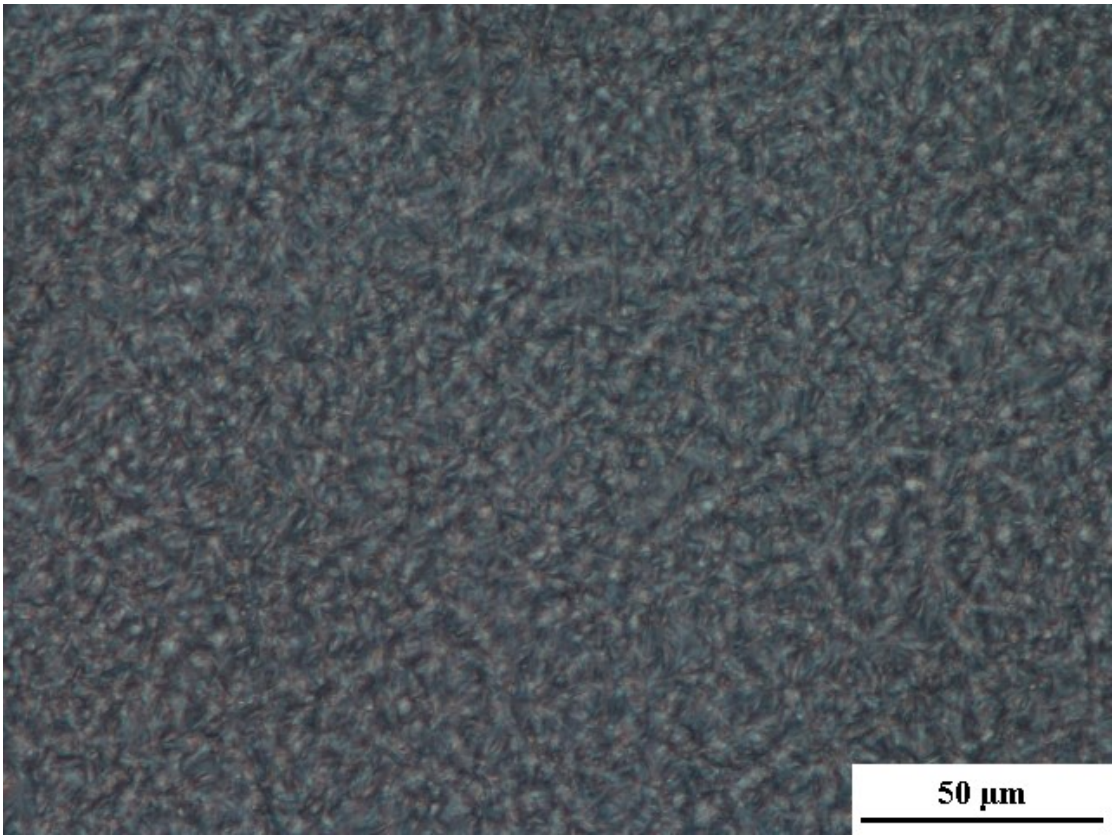

Figure S8. The 500× microphotograph of A3 sample.

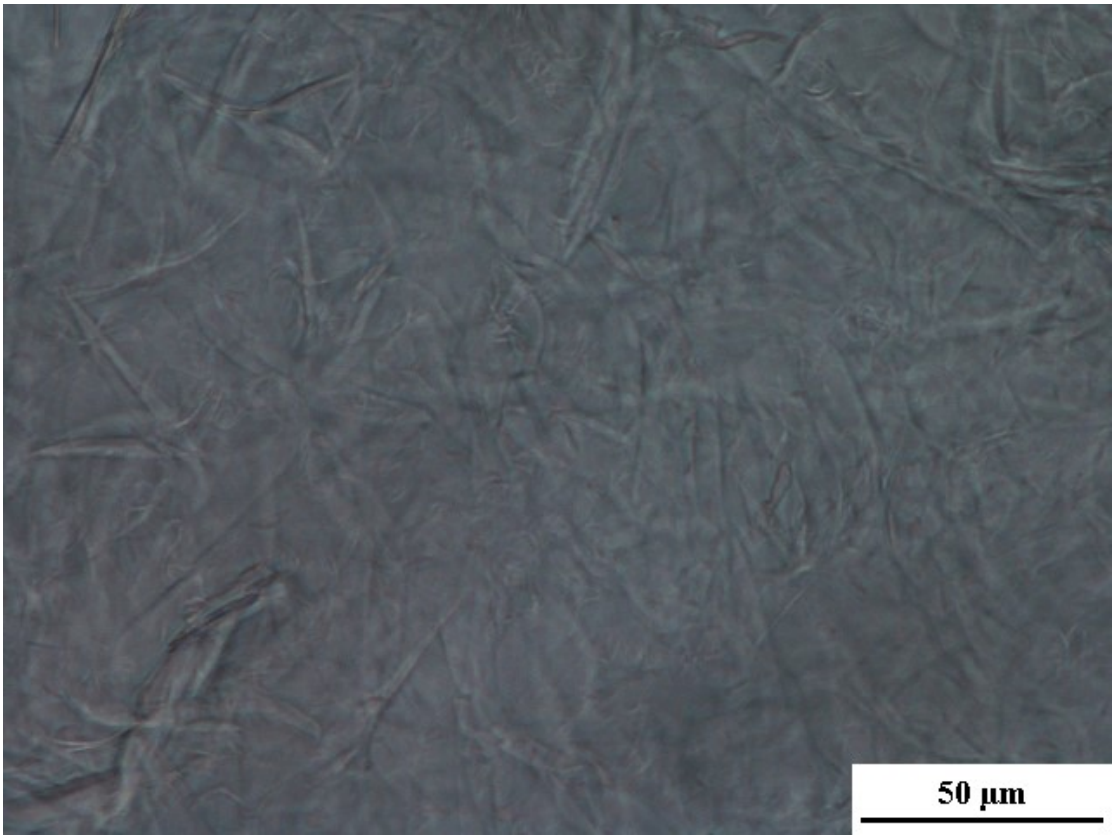

Figure S9. The 500× microphotograph of A4 sample.

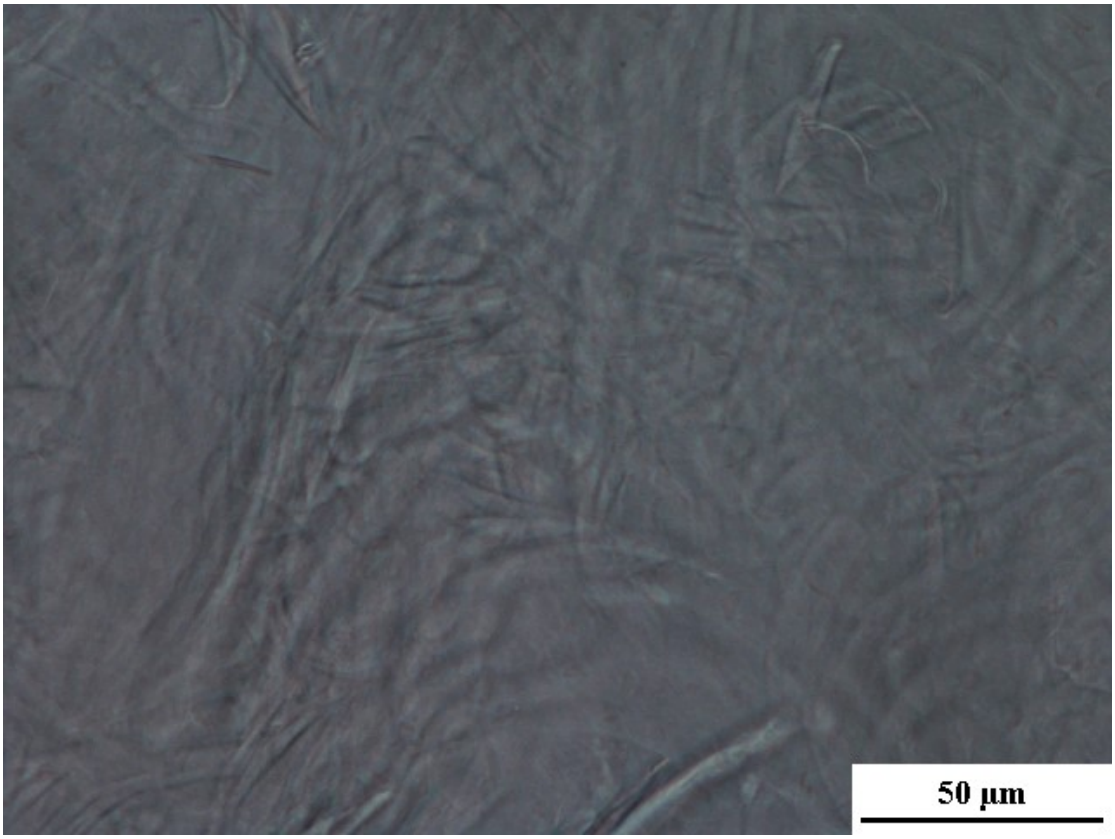

Figure S10. The 500× microphotograph of A5 sample.

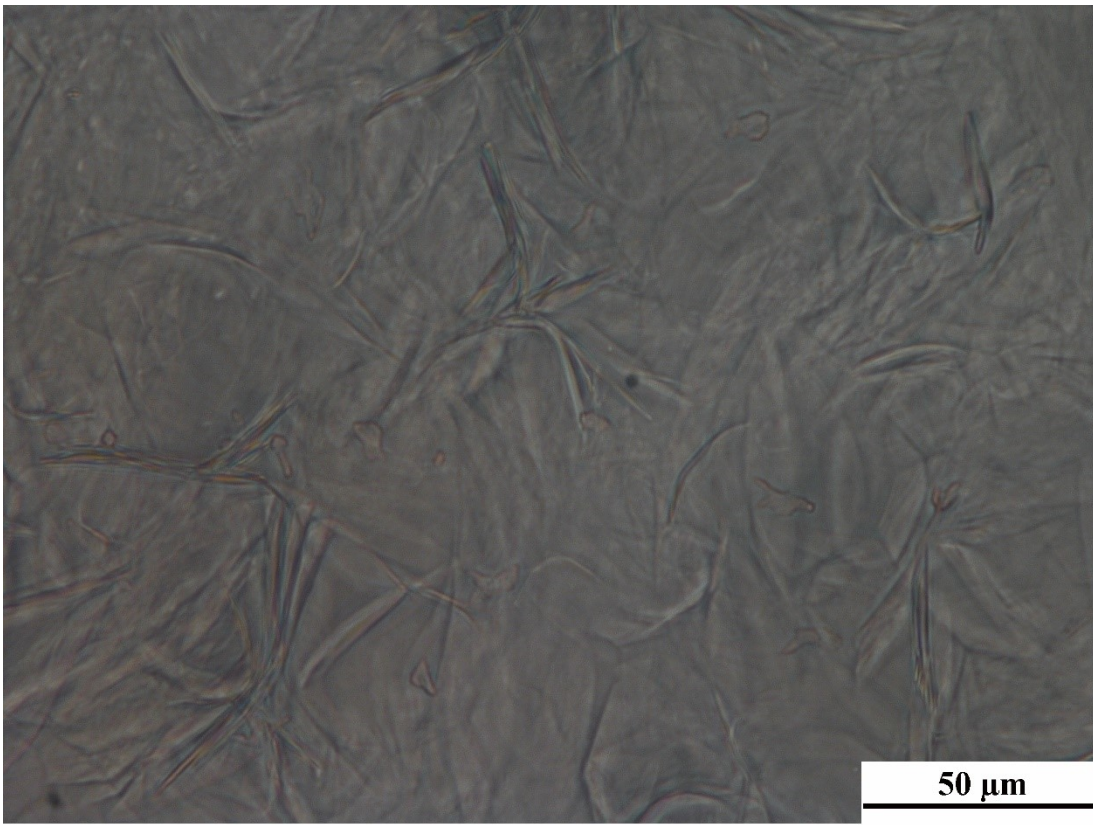

Figure S11. The 500× microphotograph of A6 sample.

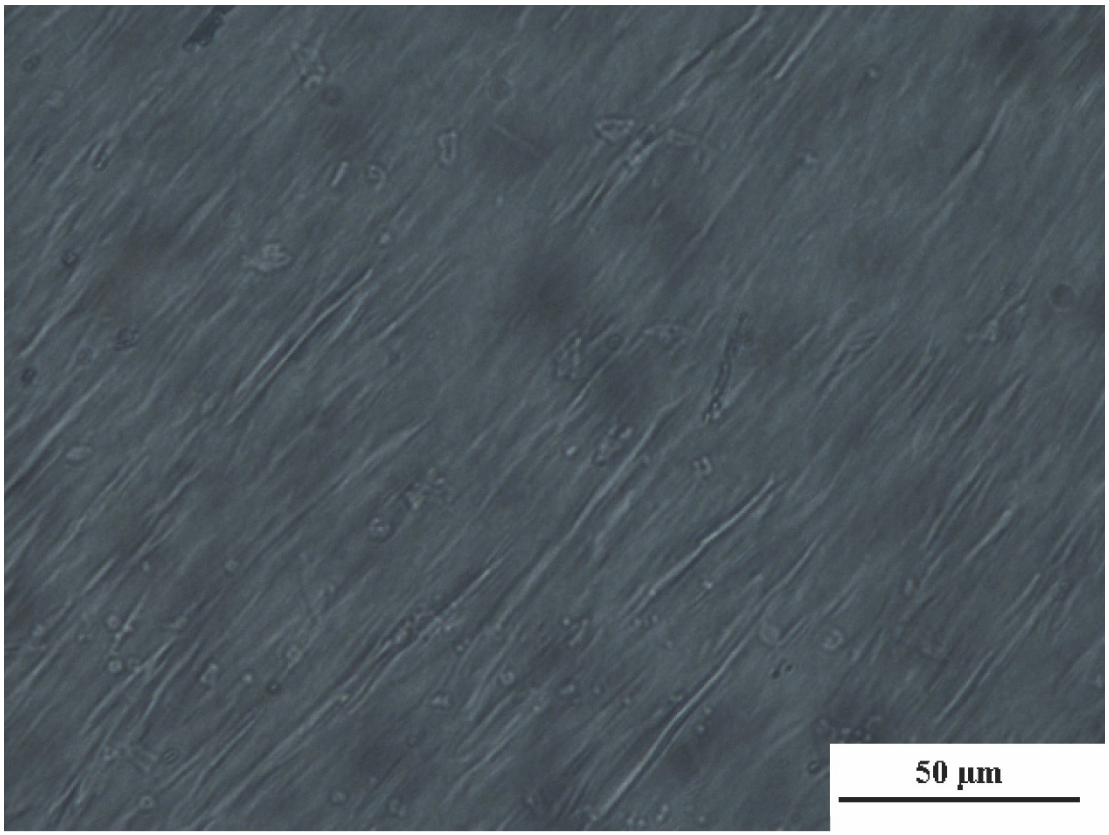

Figure S12. The 500× microphotograph of SA.

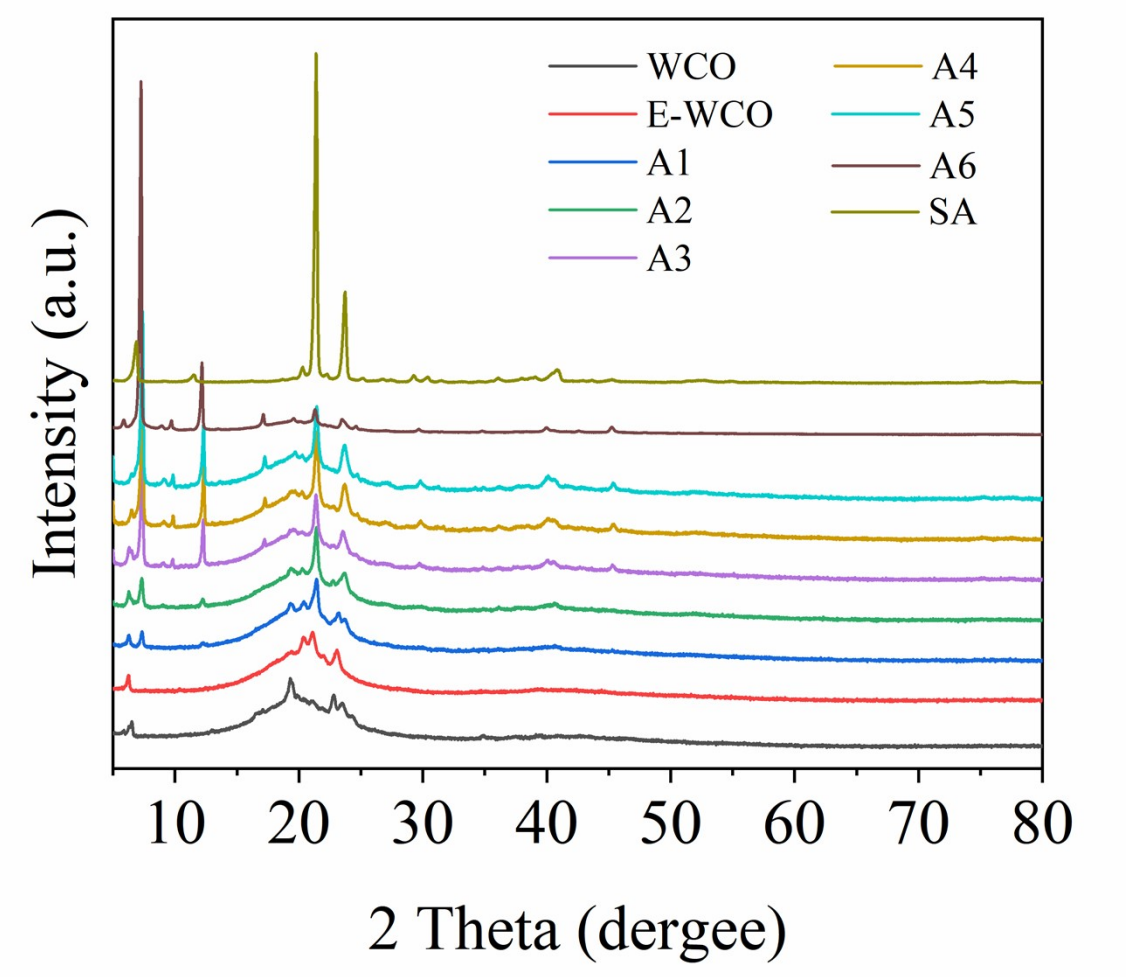

Figure S13. The PXRD patterns of WCO-based waxes and other control samples.