Response of Properties and Chemicals to Preparation Parameters in the Oxidation Processing of Refined Montan Wax

Xian Luo, Weifeng Dai, Yi Qin, Shiyun Jiao, Baocai Li, and Mi Zhang*

ABSTRACT: Refined montan wax (RMW) is a lignite-based chemical product with wide application and high added value. However, research on its processing and performance is very limited. Currently, four parameters in the key preparation procedure for the oxidation bleaching of RMW, including the concentration of two oxidants (H₂SO₄ (P₁) and CrO₃ (P₂)), oxidation time (P₃), and the mass ratio of CrO₃ used in two oxidation steps (P₄), were systematically evaluated in regard to their impact on the properties and chemistry of RMW. The results showed that the four tested parameters visibly affected RMW, and each parameter had a different impact on the properties of RMW by range analysis, of which P₁ showed a greater influence on its acid value; P₂ influenced its friability, specific surface area, and aperture; P₃ affected its color, initial melting point, and saponification value; and P₄ had a higher impact on its final melting point, melting range, and hardness. Gas chromatography with flame ionization detection–mass spectrometry analysis revealed that the compounds found in RMW samples (RMWs) under different oxidation conditions differed significantly, with major differences in the content and amount of these components. Among the compounds in RMWs, 16 different compounds (variable importance of projection > 1) were found by the orthogonal projections to latent structures discriminant analysis method, nine of which have a strong relationship to the different performances of RMWs. This work provided a basis for the development of performance-oriented preparation processing technology for RMW.

1. INTRODUCTION

Montan wax is a natural wax extracted from lignite that demonstrates good performance and wide application. Compared to synthetic or semisynthetic waxes, montan wax contains natural long-chain fatty acids, polyhydroxy fatty acid esters, and a small amount of free fatty acids, granting it a higher melting point and better stability.¹⁻⁶ Therefore, it has been considered as a potential substitute for palm wax. Currently, montan wax products include crude montan wax (CMW), deresined montan wax (DMW), and refined montan wax (RMW), which differ not only in appearance but also in performance and application. In its industrial production, CMW is directly extracted from lignite using organic solvents by continuous extraction under atmospheric pressure, which contains resin with a content of 15–30 wt %. The color of CMW is almost black and is usually used as a substitute or supplement for expensive natural animal or vegetable waxes as well as for the production of carbon paper, leather shoe polish, floor wax, glazing wax, and metal polishing agents.⁶⁻⁹ DMW is prepared from CMW through a deresination process, in which the resin content is controllable (4–15 wt %) by continuous extraction at low temperatures. The color of CMW is close to dark brown and is usually used as a raw material to prepare RMW or applied in areas that require a higher performance compared to CMW.¹⁰ RMW is prepared from DMW by oxidative bleaching, through which the color of RMW becomes close to yellow or even white and the resin content is reduced to trace amounts. Compared to CMW and DMW, RMW has
noticeable advantages in appearance for use in high-value products such as lipsticks, hair gels, creams, and precision casting. However, for this wide applicability, the requirements of the appearance and performance of MW are quite different. For example, the RMW used in lipsticks requires a harder wax than in hair gels, while a higher melting point is needed in precision casting than for creams. Precisely because of these personalized needs, the precise preparation of these materials has become an important focus of the material chemical industry. In our previous investigation, the influence on the appearance and performance of the products was of analytical grade and purchased from Tianjin Fengchuang Chemical Reagent Scientific Co., Ltd. (Tianjin, China). The oxidation bleaching process of RMW preparation from DMW involved four procedures (Figure 1): stage-one oxidation, stage-two oxidation, pickling, and water washing. The material ratio of DMW with 10 wt % residual MR/CrO3/sulfuric acid used in this reaction was 1:1.2:5.

First, 10 g of DMW was added to a 250 mL three-neck flask with a stirring device and was mixed with 50 g of sulfuric acid solution with a given concentration (P1). The reactant was heated and stirred until the sample melted. Then, a certain amount of chromium trioxide oxidant aqueous solution (P2) was slowly added to the sample through a dropping funnel at the temperature range of 105–110 °C. After reacting for several hours (P3), the sample and waste liquid were stratified, and the waste liquid in the lower layer was decanted after cooling. The procedure for stage-two oxidation was similar to that of stage-one oxidation with the exception of the mass ratio of chromium trioxide used in the two oxidation steps (P4).

Table 1. The Parameters in Oxidation Bleaching of RMWs and Their Change Levels

| variable parameters                      | level 1 (L1) | level 2 (L2) | level 3 (L3) | level 4 (L4) | invariant parameters |
|-----------------------------------------|--------------|--------------|--------------|--------------|----------------------|
| P1: concentration of H2SO4 in each stage of oxidation | 30%          | 40%          | 50%          | 60%          | 50% H2SO4 aqueous solution; 3 h; 7:3 |
| P2: concentration of CrO3 aqueous solution in each stage of oxidation | 30%          | 40%          | 50%          | 60%          | 50% H2SO4 aqueous solution; 3 h; 7:3 |
| P3: oxidation time in each stage of oxidation | 1 h          | 2 h          | 3 h          | 4 h          | 50% H2SO4; 50% CrO3 aqueous solution; 7:3 |
| P4: the mass ratio of CrO3 in two stages of oxidation | 6:4          | 7:3          | 8:2          | 4:4          | 40% H2SO4; 50% CrO3 aqueous solution; 3 h |

While oxidation processing is the most important and complex process for the preparation of RMW, how the preparation parameters affect the properties and chemistry of RMW remains unknown. Thus, the four main parameters in the oxidation bleaching of RMW, including the concentration of the two oxidants (H2SO4 (P1) and CrO3 (P2)), the oxidation time (P3), and the mass ratio of CrO3 used in two oxidation steps (P4), were evaluated systematically for their respective impact on the properties and chemistry of RMW in order to provide a basis for the development of performance-oriented preparation processing technology for RMW.

2. MATERIALS AND METHODS

2.1. Materials. Deresined montan wax (DMW) was provided by Yunnan Shangcheng Biotechnology Co., Ltd. (Yuxi, China), in which the residual resin content was 10 ± 1.2 wt %, as determined by the MW analysis method prescribed by the national standard. All chemical reagents used in this work were of analytical grade and purchased from Tianjin Fengchuang Chemical Reagent Scientific Co., Ltd. (Tianjin, China). The oxidation bleaching process of RMW preparation from DMW involved four procedures (Figure 1): stage-one oxidation, stage-two oxidation, pickling, and water washing. The material ratio of DMW with 10 wt % residual MR/CrO3/sulfuric acid used in this reaction was 1:1.2:5.

In the above-mentioned process, four parameters, including the concentration of the two oxidants (H2SO4 (P1) and CrO3 (P2)), the oxidation time (P3), and the mass ratio of CrO3 used in the two oxidation steps (P4), were adjusted. A series of RMW products were prepared under different conditions with

Figure 1. Flow chart of oxidation bleaching of RMW.
different parameters, which are specified in Table 1. In addition, the product yields of the RMW samples (RMWs) under different conditions are listed in Table S1 in the Supporting Information.

2.2. Performance Analysis of RMWs. 2.2.1. Appearance. The colors of the RMWs prepared under different conditions were observed in natural light by three staff members working in the QC department of Yunnan Shangcheng Biotechnology Co., Ltd. (Yuxi, China). To quantitatively describe the differences in appearance, the color of each RMW was scored using a color comparison ruler according to staff evaluation (Figure 2).

![Figure 2](image)

**Figure 2.** Color appearance of RMWs prepared from DMWs under different parameters in oxidation bleaching and the color comparison ruler used for quantitative description of the color.

2.2.2. Physical and Chemical Properties. The basic physical properties of RMWs under different conditions were determined in this work. The melting point was measured using a WRS-3 melting point instrument (Shanghai, China), the friability was measured with an FT-2000 friability tester (Tianjin, China), and the microhardness was determined using an HXS-1000 Vickers microhardness tester (Shanghai, China). The determination methods of the melting point, melting ranges, microhardness, and friability had been detailed in our previous publication.10

The chemical properties of RMWs under different conditions, including acid and saponification values, were determined according to the GB/T 2559-2005 method of lignite wax. The procedure was similar to the one mentioned in our previous publication.10,24

2.3. Composition Analysis by Gas Chromatography–Mass Spectrometry (GC–MS). GC–MS analysis was carried out on a GC7890B/MSS977A instrument (Agilent, Palo Alto, USA) equipped with an HP-S capillary column (30 m × 0.32 mm × 0.25 μm, Agilent). An ether solution of diazomethane used was prepared by our laboratory according to the literature.25 Bis(trimethylsilyl) trifluoroacetamide (BSTFA + TMCS) was purchased from Tokyo Chemical Industry (Tokyo, Japan). First, 4 mg of analytical-grade hexatriacontane (GC > 99%, Shanghai Aladdin Biochemical Technology Co., Ltd., Shanghai, China) was weighed and dissolved in 10 mL of toluene to prepare a 0.4 mg/mL hexatriacontane solution as the internal standard solution. Then, 5 mg of powdered RMW was placed in a 5 mL derivatization vial, in which 2 mL of a toluene solvent and 500 μL of the internal standard solution were added. Next, the cap was covered, and the vial was heated to 60 °C to dissolve the RMW. The derivatization step was carried out according to the literature.10

The GC-FID analysis conditions were as follows: the injection volume was 2 μL, the injection port pressure and temperature were 17.81 psi and 300 °C, respectively, the injection mode was splitless, and the chromatographic column had a constant flow. The carrier gas was high-purity N2 with a flow rate of 3 mL/min, the fuel gas was high-purity H2 with a flow rate of 45 mL/min, and the supporting gas was high-purity air with a flow rate of 300 mL/min. The detector temperature was set as 300 °C, and the temperature program was as follows: the initial temperature was 160 °C and maintained for 3 min, increased to 235 °C at a rate of 4 °C/min, and finally increased to 300 °C at a rate of 3 °C/min and maintained for 6 min. For MS analysis, the transmission line temperature of MS was 300 °C, and the quadrupole temperature was 150 °C. The electron energy was 70 eV, with the ion source temperature was set at 230 °C. The acquisition mode was set to full scan, and all isolated compounds were identified and analyzed by the NIST 08 database. The relative content of each component in the sample was calculated according to the following formula:

\[
\text{relative content of detected components} = \frac{A_i}{(A_2 \times C)}
\]

where \(A_i\) is the peak area of the composition to be tested, \(A_2\) is the peak area of the internal standard, and \(C\) is the internal standard concentration.

2.4. Statistical Analysis. The Excel 2019 software was used to process the experimental data of each group, and the single-factor analyses of ANOVA, LSD, and the Duncan method were performed using the SPSS statistical analysis software (p < 0.05) to analyze the significance of the difference. In addition, the influence or relationship of the oxidation parameters to the properties or chemistry of the sample was analyzed by range analysis, principal component analysis (PCA), and orthogonal projections to latent structures discriminant analysis (OPLS-DA) using the Origin 2019 software.

3. RESULTS AND DISCUSSION

3.1. Color Response to the Preparation Parameters in the Oxidation Processing of RMW. Through oxidation bleaching, the colors of the RMWs had significantly improved compared to those of DMW. However, because the parameters in the oxidation process were different, the colors of the appearance-improved RMWs also differed. The shade of the color for each RMW was quantified using numbers from low (light) to high (dark) (Figure 2 and Table S2 in the Supporting Information). Range analysis indicated that P3 had the greatest influence on the color followed by P2, P4, and P1 (Figure 3 and Table S3 in the Supporting Information). In other words, the color of the RMW was influenced by a
comprehensive effect induced by each parameter in the oxidation processing of the RMW. However, the impact of each parameter was different.

It is well-known that differences in appearance reflect the internal differences of the products, including their properties and compositions. These results revealed that the four parameters tested in the oxidation process have obvious and various effects on the color of the RMW. A prior investigation showed that the residual MR content in DMW had a positive correlation with its resulting color, as the color of the RMW was found to deepen as the residual MR content in DMW increased.10,26 In practical production, the average residual MR content in DMW is usually ~10 wt % by industrial deresination technology.10,23 Thus, the color requirement of RMW could be achieved by adjusting these four parameters in the oxidation process of RMW and optimizing them based on their influence on the color.

3.2. Effect of Preparation Parameters on the Properties of the RMWs after Oxidation Processing. Five main physical properties, including the melting point, microhardness, friability, Brunauer–Emmett–Teller (BET) surface area, and aperture, as well as two chemical properties, including acid and saponification values, were detected by their corresponding methods.27–29 As a result, these seven properties differed in various degrees for RMWs prepared under different conditions, indicating that the four parameters considered in oxidation processing affected the performance of the RMWs (Figure 4). The influence of each parameter on the properties of the oxidation processing of RMW was analyzed by range analysis and ordered as follows: for the color, P3 > P2 > P4 > P1; for the initial melting point and the saponification value, P3 > P1 > P2 > P4; for the final melting point, P4 > P1 > P2 > P3; for the melting range, P4 > P2 > P3 > P1; for the hardness, P4 > P3 > P1 > P2; for the friability and aperture P2 > P3 > P4 > P1; for the specific surface area, P2 > P3 > P1 > P4; and for the acidity, P1 > P4 > P2 > P3 (Figure 3).

Performance is the basis of material application, which can be determined and adjusted by the processing parameters. For widely used chemical products, the different applications have different requirements for RMWs. For instance, RMW added

Figure 3. Changes in physical and chemical properties of RMWs prepared from DMWs under different parameters in oxidation bleaching. (a) Initial melting point (n = 3); (b) final melting point (n = 3); (c) melting range (n = 3); (d) Vickers hardness under a 50 gf test force (n = 3); (e) friability expressed by the weight loss ratio (n = 3); (f) BET surface area (n = 1); (g) aperture (n = 1); (h) acid value (n = 3); (i) saponification value (n = 3). Different small letters in each figure indicate the significant differences among different RMW samples (p < 0.05).
to car wax requires a higher melting point than when added to floor wax, while the required hardness of RMW added to leather wax is higher than for glazing wax.30,31 On the other hand, the acidity and the saponification value are two indexes that characterize the chemical properties of RMWs.10,25 Our previous investigation showed that they strongly affected the physical properties of RMWs.10 In addition, the response of each property to each parameter in the oxidation processing of RMW showed obvious differences. Based on the influence of each parameter on the properties of RMW, the performance of RMW can be improved or optimized to meet the personalized requirements for different applications.

3.3. Chemical Response to Preparation Parameters in the Oxidation Processing of RMWs. The chemical compositions of RMWs obtained under different oxidation refining conditions were analyzed by gas chromatography–mass spectrometry (GC–MS), and the relative content of each identified chemical component in RMW was determined by GC. In total, 33 chemical substances (matching degree > 80%) were identified from these RMWs, of which 10 were shared components (Figure 5a and Table S4 in the Supporting Information). Based on the composition and content determination results, obvious differences were observed in each RMW product under different conditions (Figure 5b). PCA analysis on the composition and content of different RMWs indicated a strong positive correlation shown among RMWs prepared under all three levels of conditions of P4 variation, a strong negative correlation among RMWs prepared under three of four levels of conditions of P2 variation, an even stronger negative correlation shown among RMWs prepared under the three levels of conditions of P3 variation, and a negative correlation between RMWs prepared under the two levels of conditions of P1 variation with the other level, suggesting that the influence of each parameter in the oxidation processing of RMWs on the composition and content of each was different (Figure 5c). OPLS-DA analysis showed 16 chemicals with significantly different compositions (variable importance of projection (VIP) > 1) in different RMWs among all detected chemicals (Figure 5d), which were probably the main cause of differences in appearance and performance.

The difference in the appearance and performance of the product is caused by the different chemicals in each product, and variation in processing parameters directly leads to changes in the chemical compositions. Through instrumental analysis, determining the composition of products is often more efficient, rapid, accurate, and sensitive than performance detection. Thus, research on the chemical response to preparation parameters in the oxidation processing of RMW would provide more direct evidence regarding the appearance and performance response to preparation parameters. On the other hand, the chemical compositions of raw materials will also impact the appearance and performance of the product.10 In our previous publication, the experimental sample (DMW with about 10% residual montan resin) used in this study was
be seen that the compounds numbered C13, C15, C19, and C23 had strong positive correlations with the color, friability, and specific surface area; C5, C12, C27, and C31 had strong positive correlations with the initial melting point, hardness, aperture, and saponification value; C2, C25, and C30 had strong positive correlations with the final melting point; and C3, C9, and C22 had strong positive correlations with the melting range and acid value. Notably, among these above-mentioned related compositions, nine of them had significantly different compositions (VIP > 1), indicating the correlation between the difference in performance and the difference in chemical composition and providing some basis for the study of performance metrics based on the investigation of chemical composition.

4. CONCLUSIONS

The four parameters considered in the oxidation bleaching of RMW, i.e., the concentration of two oxidants (H2SO4 (P1) and CrO3 (P2)), the oxidation time (P3), and the mass ratio of CrO3 used in two oxidation steps (P4), had significant impacts on the color, properties, and chemistry of RMW. It was determined that P1, P2, P3, and P4 each had a respectively greater influence on the acid value; the friability, specific surface area, and aperture; the color, initial melting point, and saponification value; and the final melting point, melting range, and hardness of RMW. Furthermore, the 33 chemicals detected by GC-FID/MS analysis in the RMWs under different oxidative conditions differed significantly, in which 10 were shared components and 16 were distinct components. Among these chemicals in RMWs, nine distinct components had a strong relationship to the performance of RMW, as determined by PLS analysis.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c06650.

Product yields of RMWs under different oxidation processing conditions, data on the appearance and performance of RMWs, range analysis on the influence of each parameter in oxidation bleaching of RMWs, and compositions and relative contents of chemical compositions in RMWs (PDF)

AUTHOR INFORMATION

Corresponding Author

Mi Zhang — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China; orcid.org/0000-0001-6410-6128; Phone: +86 871 6592 0738; Email: mizhangkmust@126.com; Fax: +86 871 6592 0738

Authors

Xian Luo — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China

Weifeng Dai — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China

Yi Qin — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China

Shiyun Jiao — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China

Baocai Li — Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming, Yunnan 650500, P. R. China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.1c06650

Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

This study was supported by the National Natural Science Foundation of China (21460018).

REFERENCES

(1) Noskova, L. P. Gas-chromatographic analysis of the aliphatic fractions of brown coal wax. Solid Fuel Chem. 2010, 44, 319–323.
(2) Zhang, Z.; Qin, H.; Sun, Y.; Qian, X.; Li, B.; Jiao, S.; Zhang, M. Study on the chemical composition and quality of montan wax from lignite in Heilongjiang. Guangdong Chem. Ind. 2016, 43, 44–46.
(3) Noskova, L. P. Component composition of deresined brown coal wax. Solid Fuel Chem. 2008, 42, 320–324.
(4) Lesar, B.; Straže, A.; Humar, M. Sorption properties of wood impregnated with aqueous solution of boric acid and montan wax emulsion. J. Appl. Polym. Sci. 2011, 120, 1337–1345.
(5) Anzenberger, C.; Li, S.; Bouzidi, L.; Narine, S. S. Synthesis of waxes from vegetable oil derived self-metalsized aliphatic esters. Ind. Crops Prod. 2016, 89, 368–375.

(6) Lutz, M. Natural montan wax and its raffinates. Eur. J. Lipid Sci. Technol. 2001, 103, 239–248.

(7) Huang, Y.; Zhang, M.; Zhang, H.; Qin, Y.; Dai, W.; Li, B. Evaluation of effects of different oxidation systems on chemical modification of montan resin. J. Kunning Univ. Sci. Technol. 2017, 42, 95–98.

(8) Wang, L.; Zeng, L.; Zhang, H.; Tang, P.; Du, H.; Li, B. Oxidation refinement and removing resin of montan. Chin. J. Spectrosc. Lab. 2013, 30, 158–161.

(9) Kulkarni, M. G.; Sawant, S. B. Physico-chemical properties of wax esters synthesised from corresponding alcohols using hydrobromic acid and hydrogen peroxide action. Eur. J. Lipid Sci. Technol. 2002, 104, 387–393.

(10) Chen, Y.; Zhang, H.; Dai, W.; Xian, C.; Jiao, S.; Li, B.; Zhang, M. The influence of montan resin in crude montan wax on the character and performance of refined montan wax. Energy Fuels 2020, 34, 3614–3620.

(11) Yuan, C.; Qin, Y.; Zhang, M.; Zhang, H.; Jiao, S.; Li, B. A new method of testing and evaluating the quality of refined montan wax: digital color and GC fingerprint. Chromatographia 2015, 78, 1283–1292.

(12) Wang, Y.; Herdegen, V.; Repke, J.-U. A model approach for the montan wax extraction: model development and experimental analysis. Sep. Sci. Technol. 2015, 50, 2311–2326.

(13) Kamairudin, N.; Gani, S. S.; Masoumi, H. R.; Hashim, P. Optimization of natural lipstick formulation based on pitaya (Hylocereus polyrhizus) seed oil using D-optimal mixture experimental design. Molecules 2014, 19, 16672–16683.

(14) McIntosh, K.; Smith, A.; Young, L. K.; Leitch, M. A.; Tiwari, A. K.; Reddy, C. M.; O’Neil, G. W.; Liberator, M. W.; Chandler, M.; Baki, G. Alkenones as a promising green alternative for waxes in cosmetics and personal care products. Cosmetics 2018, 5, 34.

(15) Rubiano, S.; Echeverri, J. D.; Salamanca, C. H. Solid lipid nanoparticles (SLNs) with potential as cosmetic hair formulations made from Otoba wax and ultrahigh pressure homogenization. Cosmetics 2020, 7, 42.

(16) Yu, J.; Shi, Y.; Ji, X.; Hong, Y.; Su, Y.; Su, Z. Optimal design for mid temperature pattern wax modified by EVA. Hot Work. Technol. 2013, 42, 35–36.

(17) Pan, S.; Sivanathan, S.; Kiepe, G.; Kiepe, T.; Germann, N. Candidate formulations for a sustainable lipstick supplemented with Vitamin D3: effects of wax type and concentration on material properties. Ind. Eng. Chem. Res. 2021, 60, 2027–2040.

(18) Tan, S.; Hao, X.; Wang, D.; Lei, S.; Jiang, M.; Shu, D. Numerical simulation and intelligent optimization of wax injection in investment casting. J. Phys.: Conf. Ser. 2020, 1653, No. 012016.

(19) Chen, Y. The influence of montan resin on the character and performance of montan wax. Kunming University of Science and Technology, Energy Fuels, 2020, 34(3), 3614–3620.

(20) Zhang, S.; Liu, J.; Kan, M.; Jiang, Y. Lignite wax industrial preparation and purification technology. Chem. Ind. Eng. Prog. 2011, 30, 509–513.

(21) Yuan, C.; Zhang, H.; Zhang, M.; Wei, X.; Li, B. Environment friendly bleaching methods of montan wax. J. Chem. Pharm. Res. 2014, 6, 1223–1229.

(22) Tang, P.; Zhang, H.; Xiang, C.; Wang, L.; Li, B. Regenerate Cr(VI) oxidation bleaching solution from wastewater in process of oxidation bleaching of montan wax by indirect electrochemical oxidation. Asian J. Chem. 2015, 27, 625–629.

(23) He, L.; Bian, Z.; Li, M.; Jiang, G.; Li, X.; Lui, C. Research progress in extraction and purification of lignite wax. Shandong Chem. Ind. 2019, 48, 61–62.

(24) Yuan, C.; Zhang, H.; Zhang, M.; Wei, X.; Li, B. New oxidation bleaching technology of deresinated montan wax. Chem. Ind. For. Prod. 2015, 35, 97–104.

(25) Lanzhou University Organic Chemistry Experiments. Higher Education Press Beijing, 2010.

(26) Zhu, J.; Zhang, S.; Guo, B. The study of obtaining lignite wax from lignite with petroleum ether as a solvent. Shandong Chem. Ind. 2014, 43, 39–41, 44.

(27) Zhao, P.; Liu, H.; Li, S.; Lin, H.; Jia, Y.; Yan, M.; Yuan, M.; Lin, J. Experimental investigation of the adsorption characteristics of mixed coal and variations of specific surface areas before and after CH4 adsorption. Appl. Sci. 2019, 9, 524.

(28) Boruah, A.; Rasheed, A.; Mendhe, V. A.; Ganapathi, S. Specific surface area and pore size distribution in gas shales of Raniganj Basin, India. J. Pet. Explor. Prod. Technol. 2018, 9, 1041–1050.

(29) Kwon, S.; Hwang, H.; Lee, Y. Effect of pressure treatment on the specific surface area in Kaolin group minerals. Crystals 2019, 9, 528.

(30) Zhang, J.; Man, J.; Feng, Y.; Yang, S.; Feng, Q.; Kan, D. Developing status of waxes for leather. Leath. Chem. 2011, 28, 29–32.

(31) Merusi, F.; Filippi, S.; Polacco, G. Effect of synthetic and functionalized waxes on bituminous binders: From the glassy state to the intermediate viscoelastic domain. Constr. Build. Mater. 2017, 136, 541–555.

(32) Chen, Y.; Zhang, H.; Luo, X.; Jiao, S.; Li, B.; Zhang, M. Influence of the processing factors on deresination rate and the residual resin on properties of deresined montan wax. Chem. Eng. Commun. 2021, 1–10.