Dear Editor and Reviewers:

Thank you for your letter and for the reviewers' comments concerning our manuscript entitled "The influence of various solvents extraction on chemical properties on Chang 7 shale, Ordos Basin, China" (ID: egusphere-2023-272). Those comments are all valuable and very helpful for improving our paper. We have studied comments carefully and have revised the manuscript which we hope meet with approval. Point-by-point responses to the nice reviewer are listed below this letter.

Responds to the RC1's comments:

RC1: The manuscript combines infrared spectroscopy with Soxhlet extraction to unveil the variations in the chemical properties of different maturity organic-rich lacustrine shales before and after solvent extraction. The article is well-written, and the research findings are valuable for guiding the selection of organic solvents in oil shale washing experiments involving different maturity lacustrine shales. However, there are a few questions that require clarification from the authors, which I consider to be a minor revision.

Response: Thank you very much for your valuable suggestions. We have revised the content of the manuscript and enriched the submitted manuscript according to the reviewers re quirements. Those suggestions were helpful and significantly improved the quality of our article.

Detailed comments:

Point 1: During the extraction process, the samples were ground to 60 mesh. Please provide an explanation for this step.

Response 1: Explanation and revise

Thank you for pointing this out. The bulk shale samples were uniformly crushed into 60 mesh with a laboratory knife mill and then evenly divided into five parts. (See lines 84 and 85). In this experiment, the samples were ground to a particle size of 60 mesh for the convenience of subsequent adsorption experiments. Indeed, the particle size impacts of CO_2 and N_2 adsorption experiments is substantial. Han et al. (2020) revealed the pore and fractal characteristics of lacustrine shales of Qingshankou Formation in the Songliao Basin, northeast China by 60 mesh shale of nitrogen adsorption and mercury injection methods. In addition, Wei et al. (2014) indicated that a grain size of 60-mesh is beneficial for efficient solvent extraction of soluble OM and is more appropriate for gas adsorption porosimetry for both unextracted and extracted shales. Therefore, we took the 60 mesh as the optimal particle size for the shale analysis in this research. Also, all samples were ground to 60 mesh, and the results of those tests are comparable.

Han H, Guo C, Zhong N, et al. A study on fractal characteristics of lacustrine shales of Qingshankou Formation in the Songliao Basin, northeast China using nitrogen adsorption and mercury injection methods[J]. Journal of Petroleum Science and Engineering, 2020, 193: 107378.

Wei, L., Mastalerz, M., Schimmelmann, A., Chen, Y., 2014. Influence of Soxhlet-extractable bitumen and oil on porosity in thermally maturing organic-rich shales. Int.

J. Coal Geol. 132, 38–50.

Point 2: The objective of the manuscript is to investigate the alterations in the chemical properties of shales before and after extraction. Could you clarify the necessity of conducting XRD measurements? The authors should provide an explanation.
Response 2: Explanation
Thank you for pointing out this issue. This study is a methodological exploration, and the revelation of the samples mineral composition through XRD is of significant reference value for other readers applying the method proposed in this article. The mineral composition is an important parameter of shale, and therefore, XRD testing is necessary in this study.

Responds to the RC2's comments:
RC2: This study employed four solvents, namely acetone, tetrahydrofuran (THF), carbon disulfide (CS2), and benzene, to extract lacustrine shales of different maturities. Fourier-transform infrared spectroscopy (FTIR) was utilized to analyze the functional groups in the samples before and after solvent extraction. The experimental design by the authors is sufficiently innovative, and the manuscript presents convincing data and precise language expression. The research findings can provide valuable guidance for the selection of solvents in laboratory shale extraction and the development of shale oil in the future. However, I still have a few questions that I would like the authors to address. I recommend minor revisions for the publication of this manuscript.
Response: Thank you very much for your valuable suggestions. We have revised the content of the manuscript and enriched the submitted manuscript according to the reviewers requirements.

Detailed comments:
Point 1: Could the authors explain why these four solvents were chosen?
Response 1: Explanation and revise
Thank you for pointing this out. During the development of shale oil, the chemical properties of shale after being extracted by using different organic solvents are also different (Cao et al., 2020). The study of the changes in chemical properties in lacustrine organic-rich shale of different maturity before and after extraction with different solvents is rarely reported. Thus, the sample was extracted using solvents of different aromaticity, polarity, and permeability, namely acetone, tetrahydrofuran, carbon disulfide, and benzene, and the remaining original sample was used as a control (See lines 85 and 86). Our results of the study will help in the selection of organic solvents for oil-washing experiments in shales of different maturity.

Cao, Y., Han, H., Guo, C., Pang, P., Ding, Z. gang, and Gao, Y.: Influence of extractable organic matters on pore structure and its evolution of Chang 7 member shales in the Ordos Basin, China: Implications from extractions using various solvents, J. Nat. Gas Sci. Eng., 79, 103370, https://doi.org/10.1016/j.jngse.2020.103370, 2020.

Point 2: While FTIR was used to analyze the chemical properties of the shale, did the authors consider any other testing methods?

Response 2: Explanation

Thank you for highlighting this issue. There are several methods available for testing the chemical properties of the sample, including infrared spectroscopy, nuclear magnetic resonance, Raman spectroscopy, and others. In this study, we specifically employed the FTIR method to showcase the chemical properties of the sample before and after extraction. We believe that the research findings are sufficiently compelling to pique the interest of readers in related fields. We also appreciate the suggestions provided by the reviewer, and in future studies, we will further consider the application of multiple characterization techniques for assessing the chemical properties of shale samples.

Point 3: Why were two different figures, Figure 4a and 4b, used to present the extraction yields? What is the difference between them?

Response 3: Explanation

Thank you for pointing this out. The organic solvent extraction efficiency of shale is not only influenced by the properties of the solvents but also by the Total Organic Carbon (TOC) content of the samples. Presenting both the absolute extraction efficiency and the TOC-normalized extraction efficiency allows for a more objective reflection of the solvents extraction capabilities.

Responds to the CC1's comments:

CC1: This paper focuses on exploring the influence of various solvents extraction on chemical properties of shales. It is a topic of interest to the researchers in the fossil energy areas, the manuscript is well written. Moreover, this well-designed research paper contains interesting results which merit publication, the chemical characterization of shales with different maturities is attractive. For the benefit of the readers, however, there are still some problems need be clarified after reading this paper. I suggest some minor modifications before publication and I would be happy to review the revised article.

1: You took the 60 mesh as the optimal particle size in extraction process of shale. However, some groups have suggested 200 mesh for the shale extraction. This analytical condition could have substantial influences on subsequent shale characterization.

2: The conclusions of this paper don't refer to the mineral composition of shale. Is XRD experiment redundant in your research?

Response 1: Thank you for pointing this out. Our research is a continuation of previous research and previous work mainly evaluated the changes in pore structure of shale before and after solvent extraction using N2 sorption. Coincidentally, Wei et al. (2014) indicated that a grain size of 60-mesh is beneficial for efficient solvent extraction of soluble OM and is more appropriate for gas adsorption porosimetry for both unextracted and extracted shales. Therefore, we took the 60 mesh as the optimal particle size for the shale analysis in this research.

Wei, L., Mastalerz, M., Schimmelmann, A., Chen, Y., 2014. Influence of Soxhlet-extractable bitumen and oil on porosity in thermally maturing organic-rich shales. Int. J. Coal Geol. 132, 38–50.

Response 2: Thank you for pointing this out. XRD experiments of shale samples are necessary and the FTIR results of those samples can provide a reference for shale samples of similar mineral composition.

Responds to the CC2's comments:

CC2: The author employed four polar solvents (acetone, tetrahydrofuran (THF), carbon disulfide (CS2), and benzene) for extracting lake-type shales at different maturity levels. The extraction yield of various solvents were compared, while Fourier-transform infrared spectroscopy (FTIR) was used to examine functional group characteristics pre- and post-extraction. The experimental design was intriguing, demanding significant effort, and yielded compelling results. However, there are still unresolved inquiries that require clarification from the author. I suggest considering minor revisions to the article.

1. I would like to suggest that the TOC should be written in full name where this word appeared for the first time in the manuscript.
2. The calculation of extraction yields involves the quality of the residual sample. How to judge the effect of extraction, and is it enough to extract 150 hours?

Response 1: Thank you for pointing this out. The TOC has been written in full name where this word appeared for the first time in the manuscript (see lines 14 and 15).

Response 2: Thank you for pointing this out. In our study, the time of the experiment is constant, each studied sample was extracted for 150 hours. In previous studies of this kind, shale samples were extracted for 72 hours (Wei et al., 2014; Guo et al., 2014; Furmann et al., 2013). Although we cannot be sure that the shale-soluble organic matter has been completely extracted, we believe that 150 hours is close to the time when the organic matter has been completely extracted.

Wei, L., Mastalerz, M., Schimmelmann, A., Chen, Y., 2014. Influence of Soxhlet-extractable bitumen and oil on porosity in thermally maturing organic-rich shales. Int. J. Coal Geol. 132, 38–50.
Guo, H., Jia, W., Peng, P., Lei, Y., Luo, X., Cheng, M., Wang, X., Zhang, L., Jiang, C., 2014. The composition and its impact on the methane sorption of lacustrine shales from the Upper Triassic Yanchang Formation, Ordos Basin, China. Mar. Petrol. Geol. 57, 509–520.
Furmann, A., Mastalerz, M., Brassell, S. C., Schimmelmann, A., Picardal, F., 2013. Extractability of biomarkers from high- and low-vitrinite coals and its effect on the porosity of coal. Int. J. Coal. Geol. 107, 141–151.

Responds to the CC3's comments:

CC3: The author employed four polar solvents (acetone, tetrahydrofuran (THF), carbon disulfide (CS2), and benzene) for extracting lake-type shales at different maturity levels. The extraction yield of various solvents were compared, while Fourier-transform infrared spectroscopy (FTIR) was used to examine functional group characteristics pre- and post-extraction. The experimental design was intriguing, demanding significant effort, and yielded compelling results. However, there are still unresolved inquiries that require clarification from the author. I suggest considering minor revisions to the article.

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Response 1: Thank you for pointing this out. The TOC has been written in full name where this word appeared for the first time in the manuscript (see lines 14 and 15).

Response 2: Thank you for pointing this out. In our study, the time of the experiment is constant, each studied sample was extracted for 150 hours. In previous studies of this kind, shale samples were extracted for 72 hours (Wei et al., 2014; Guo et al., 2014; Furmann et al., 2013). Although we cannot be sure that the shale-soluble organic matter has been completely extracted, we believe that 150 hours is close to the time when the organic matter has been completely extracted.

Wei, L., Mastalerz, M., Schimmelmann, A., Chen, Y., 2014. Influence of Soxhlet-extractable bitumen and oil on porosity in thermally maturing organic-rich shales. Int. J. Coal Geol. 132, 38–50.

Guo, H., Jia, W., Peng, P., Lei, Y., Luo, X., Cheng, M., Wang, X., Zhang, L., Jiang, C., 2014. The composition and its impact on the methane sorption of lacustrine shales from the Upper Triassic Yanchang Formation, Ordos Basin, China. Mar. Petrol. Geol. 57, 509–520.

Furmann, A., Mastalerz, M., Brassell, S. C., Schimmelmann, A., Picardal, F., 2013. Extractability of biomarkers from high- and low-vitrinite coals and its effect on the porosity of coal. Int. J. Coal. Geol. 107, 141–151.

Responds to the CC4's comments:

CC4: The experiments seem well-designed, the data are convincing, and the entire article is clear and scholarly. The specific comments, which have to be addressed before the article can be recommended for publication are:

1. The OM should be written in full name where this word appeared for the first time in the manuscript.
2. The discussion regarding the extraction of organic matter may be strengthened by the use of NMR analyses of the samples if available.

Response 1: Thank you for pointing this out. The OM has been written in full name where this word appeared for the first time in the manuscript (see line 17).

Response 2: Thank you for pointing this out. It is our negligence of our experimental design and we are sorry about this. We agree the reviewer's point. Strictly speaking, the discussion regarding the extraction of organic matter may be strengthen by use of NMR analyses of the samples in addition to FTIR if available. Althrough Both Nuclear Magnetic Resonance (NMR) and Infrared Spectroscopy contribute to our understanding of shales organic properties to some extent, this study primarily focuses on discussing the results obtained from infrared spectroscopy testing, which provides insights into the organic features of shale. In addition, there are no samples left for this experiment, so we cannot do the experiment suggested by reviewers. Furthermore, the reviews comments about the use of NMR analyses of the samples would be a new direction for our next phase of research.

Conclusion: Thanks again to the reviewer for their noble opinions on the manuscript and the editor for his hard work on the manuscript. We have made detailed modifications according to the opinions of reviewer. The added references have been appropriately included in the corresponding positions within the article, as per the requested format.