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EFFECTS OF PRETREATMENT ON LASER DIFFRACTION PARTICLE SIZE ANALYSIS OF FLUVIAL SEDIMENTS IN DESERT REGIONS

Mengmeng Tang[1,2,3], Keri Yao[1,2,3], Jing[1] Meng[1,2,3], Hanxiao Guo[1,2,3], Haoran Yuan[1,2,3], Zhongyi Hu[1,2,3], Abdureyim Anwar[2,3,4], Yue Dai[1,2,3*], Feng Zhang[1,2,3*]
[1]College of Geography and Remote Sensing Sciences, Xinjiang University, Urumqi 830046, China;
[2]Xinjiang Field Scientific Observation and Research Station for the Oasisization Process in the Hinterland of the Taklamakan Desert, Yutian, Xinjiang 848400, China;
[3]Xinjiang Key Laboratory of Oasis Ecology, Urumqi 830046, China;
[4]College of Ecology and Environment, Xinjiang University, Urumqi 830046, China;
* Corresponding authors e-mail: daiyue@xju.edu.cn; zhangfeng@xju.edu.cn

Abstract: The choice of pretreatment methods in laser diffraction analysis of mineralogical grain size composition in fluvial sediments can significantly influence measurement outcomes, yet published data on this topic remain limited. This study systematically investigates the effects of four different pretreatment methods—sieving only, physical dispersion only, removal of organic matter only, and combined removal of carbonate and organic matter—on grain size measurements, using typical fluvial sediments from the terminal oasis of the Keriya River in the central Tarim Basin, Taklamakan Desert. Results showed that samples treated by sieving alone tend to yield overestimated median grain sizes, with particle size variation rates exceeding 70% in some cases. We concluded that carbonate acts as the primary cementing agent between sediment particles in this region, and acid digestion plays a crucial role in disaggregating micro-aggregates and releasing fine-grained fractions. Physical dispersion is mainly effective in breaking down loosely structured macro-aggregates. Furthermore, the removal of organic matter is essential for eliminating organic cementation and minimizing interference from water absorption and swelling in certain samples.

Keywords: grain size analysis; pretreatment methods; fluvial sediments; the Taklamakan Desert.

1. Introduction

The mechanical composition of mineral particles in soil texture is a key characteristic of soil physical properties and significantly influences soil moisture content, fertility, and other attributes. Furthermore, grain-size analysis has been widely applied in Quaternary environmental studies as an essential tool for investigating loess-paleosol sequences and reconstructing paleoenvironments and paleoclimates [1–6]. To accurately determine the mechanical composition of mineral particles, proper pretreatment prior to measurement is crucial. Conventional pretreatment procedures typically involve the addition of hydrogen peroxide to remove organic matter, hydrochloric acid to dissolve carbonates, and sodium hexametaphosphate to achieve physical dispersion of particles.

Pretreatment methods vary depending on disciplinary focus and the nature of samples from different depositional environments. In paleosols developed in regions such as the Loess Plateau or similar settings, where pedogenic intensity is relatively low, applying the same pretreatment protocols used for loess samples generally yields reliable grain-size data [7–13]. However, in subtropical and tropical regions, paleosols derived from weathered eolian loess often exhibit significantly increased clay content and stronger aggregation. Applying standard loess pretreatment methods to these soils may fail to fully disperse the particles, resulting in an overestimation of coarse fractions. Some studies suggest that eolian red clays should not be treated using the same procedures as typical loess deposits [14]. Wang et al [1] have demonstrated that pretreatment methods can significantly affect grain-size measurements of lacustrine sediments. Nevertheless, optimal pretreatment strategies for arid and semi-arid region sediments remain inadequately explored. Studies by Fisher et al [15] and Beuselinck et al [16] indicated that incomplete physical or chemical dispersion of arid and semi-arid sediments can lead to the persistence of aggregates, causing distortions in optical model inversions and consequently misrepresenting the proportions of clay and silt fractions. On the other hand, considering the high salinity and carbonate content typical of arid-zone soils, Goossens et al [17] argued that carbonates often act as cementing agents stabilizing particle frameworks; conventional acid digestion and chemical dispersion steps may dissolve these cements, leading to the breakdown of natural aggregates and introducing artificial fining artifacts. Moreover, fluvial deposits in desert environments possess mixed aeolian and fluvial characteristics, with more complex mineralogical compositions and binding mechanisms. Therefore, appropriate measurement protocols must be carefully selected according to specific research objectives.

Currently, research on grain-size analysis of desert and desert river sediments has primarily focused on regional characteristics and their environmental implications, while systematic investigations into the effects of different pretreatment methods on laser diffraction-based grain-size measurements remain limited. In light of this gap, the present study systematically compares the impacts of various pretreatment methods on laser diffraction results for river sediments, aiming to provide experimental evidence and guidance for selecting appropriate protocols in determining the mechanical composition of mineral particles in arid-region fluvial deposits.

2. Materials and methods

2.1 Study Area

The Keriya River flows northward into the terminal oasis of the Daliyabuyi in the Taklamakan Desert [18]. This oasis experiences minimal anthropogenic disturbance and largely retains its natural, relatively "pristine" condition [19]. The annual precipitation in Daliyabuyi is less than 50 mm, with a mean annual temperature of approximately 11.8 °C [20]. The river is primarily fed by snow and glacial meltwater from the Kunlun Mountains, with an annual runoff of about 9×10^8 m^3, exhibiting two flood seasons and two dry seasons in spring and summer (data from the Hotan Hydrological Bureau, Xinjiang). Dominant plant species include *Populus euphratica*, *P. schrenkiana*, *Tamarix* spp., and *Phragmites australis* [21]. From south to north across the oasis, groundwater depth gradually increases, ranging from 2 m to 9 m, and groundwater salinity varies between 4 g/L to 6 g/L. The soil pH ranges from 8.49 to 9.15, indicating alkaline conditions [19, 22–23].

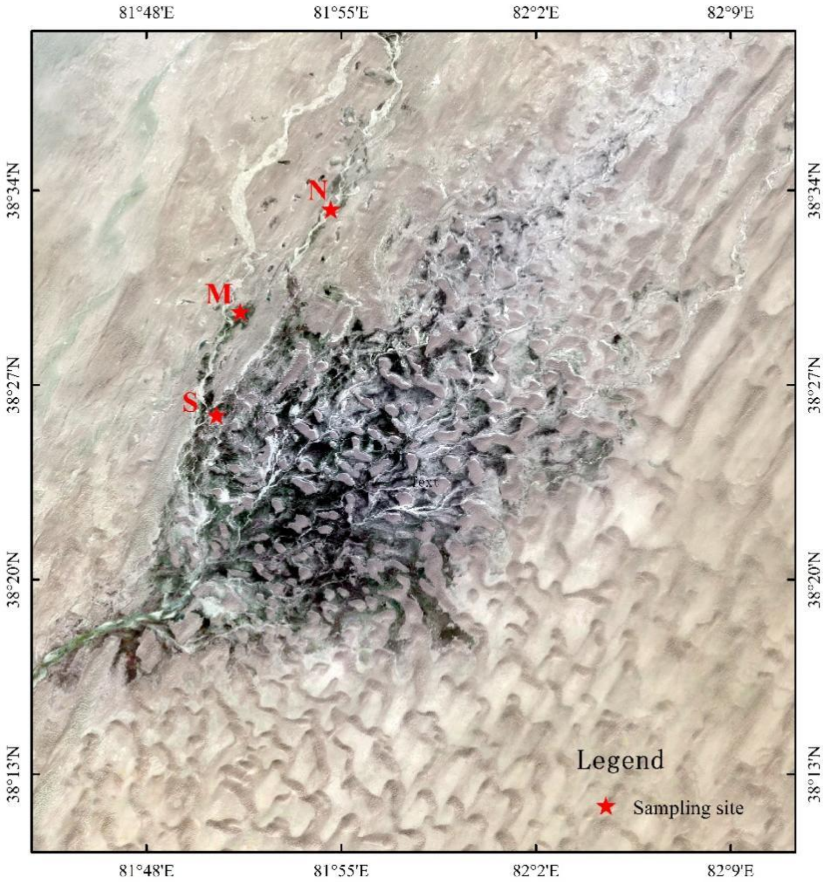

Fig. 1. Geographic location of the terminal area of the Keriya River and the distribution of sampling sites. The labels S, M, and N represent sampling sites in the southern, middle, and northern parts of the study area, respectively.

2.2 Sample collection

In May 2025, three sampling sites—S, M, and N—were selected on the western bank of the terminal reach of the Keriya River, within a distance of 200–800 m from the river channel (Fig. 1). At each site, a soil pit measuring 1 m × 1 m × 1 m was excavated. Soil samples were collected at a depth of 30 cm using a cutting ring from three vertical profiles within each pit, yielding a total of nine soil samples. The samples were sealed in self-closing bags and transported to the laboratory for subsequent testing and analysis.

2.3 Experimental methods

(1) Pretreatment of sieving only: Samples were passed through a 900 μm sieve and then directly analyzed by instrument.

(2) Pretreatment of physical dispersion: 0.08–0.09 g of each sample was weighed into a beaker, and 10 mL of 0.05 mol/L sodium hexametaphosphate ($(NaPO_3)_6$) dispersant was added. The mixture was ultrasonicated at a frequency of 40 kHz for 15 min before instrumental analysis.

(3) Pretreatment of removal of organic matter followed by physical dispersion: 0.08–0.09 g of each sample was treated with 10 mL of 30% hydrogen peroxide (H_2O_2) and heated in a water bath on a hot plate set to 200 °C to remove organic matter. The reaction was maintained for 2–3 h until no further bubbling occurred. The suspension was then centrifuged after adding deionized water. After confirming that the supernatant was clear and the solids had settled tightly at the bottom of the tube,

the supernatant was discarded. This washing step was repeated twice with deionized water. The final pH of the residue was tested to ensure neutrality. Subsequently, 10 mL of 0.05 mol/L $(NaPO_3)_6$ dispersant was added, and the sample was ultrasonicated at 40 kHz for 15 min prior to measurement.

(4) Pretreatment of removal of carbonates and organic matter followed by physical dispersion (Complete pretreatment): Approximately 5 g of each sample was treated with a sufficient amount of 10% HCl, heated in a water bath on a hot plate set to 200 °C to dissolve carbonate cements. The reaction proceeded for 2–3 h until gas evolution ceased. The sample was then centrifuged after addition of deionized water, and the supernatant was discarded once clarity and complete sedimentation were confirmed. This washing process was repeated three times, ensuring neutral pH after the final wash. Next, a sufficient amount of 30% H_2O_2 was added and heated in a water bath on a hot plate set to 200 °C to remove organic matter, again reacting for 2–3 h until no bubbles formed. The sample was centrifuged and washed three more times with deionized water until neutral pH was achieved. Finally, 10 mL of 0.05 mol/L $(NaPO_3)_6$ dispersant was added, and the sample was ultrasonicated at 40 kHz for 15 min before instrumental analysis. Each sample was measured in triplicate.

Grain size analysis was conducted using a Malvern Mastersizer 3000 laser diffraction particle size analyzer at the Laboratory of Sedimentary Environments, Key Laboratory of Oasis Ecology, Xinjiang University.

3. Results

3.1 The effects of different pretreatments on grain size frequency distributions

3.1.1 Pretreatment of sieving only

Both S1 and M1 samples exhibited multimodal distributions, with peaks in the coarse fraction. The untreated S1 sample showed a distinct bimodal pattern, with a broad primary peak between 1–100 µm and a pronounced secondary peak above 1000 µm, indicating the presence of loosely aggregated coarse particles formed by fine-grained flocculation. The M1 sample displayed two peaks at 1–10 µm and 30–100 µm, respectively, with a relatively flat trough between 10–30 µm.

3.1.2 Pretreatments of physical dispersion with $(NaPO_3)_6$

For the S1 sample, the coarse peak above 1000 µm disappeared after physical dispersion, and the main peak shifted significantly toward finer grain sizes. This indicates that physical dispersion effectively disrupted weakly bound coarse aggregates in the sediment, eliminating part of the apparent coarseness. In contrast, after treatment with $(NaPO_3)_6$, the M1 sample exhibited a dominant peak between 1–60 µm, with a rightward shift and relative enhancement of peaks in the >60 µm range. This suggests that the apparent coarseness in M1 is likely due to more tightly cemented micro-aggregates; physical dispersion alone has limited effectiveness and may even induce hydration swelling of aggregates, leading to an increase in measured particle size.

3.1.3 Pretreatments of physical dispersion following organic matter removal

The variation in the grain size distribution curve of S1 was relatively mild, consistent with the curve shape from the $(NaPO_3)_6$ dispersion-only treatment. It was manifested as the main peak slightly narrowing, increasing in height, and shifting toward the fine end; this phenomenon aligns with the characteristic of low organic matter content in desert river sediments. The changes in sample M1 were more significant: the fine particle component increased, and the main peak shifted to the left and appeared narrower and taller, indicating that its aggregation mechanism is controlled by organic cementation. An obvious secondary peak remained in the range greater than 60 µm, indicating that removing organic matter alone cannot completely disperse all composite particles, and cemented aggregates are still retained in the sample.

3.1.4 Complete pretreatment

After complete pretreatment, a significant shift toward finer grain sizes was observed compared to samples treated only with H_2O_2. Both S1 and M1 exhibited stable main peaks around 10 μm (Fig. 2). This confirms that the acid digestion step effectively liberated fine particles bound by carbonate cements, thereby eliminating the final sources of spurious coarseness in the sediment.

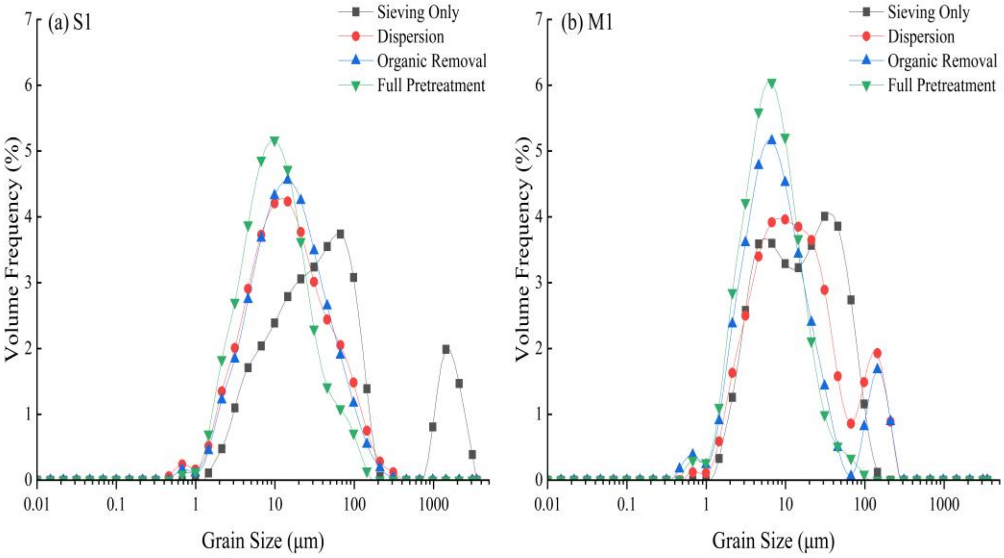

Fig. 2. Grain-size frequency distribution curves of fluvial sediments in desert regions under stepwise pretreatment conditions

3.2 Comparison of grain size parameters before and after complete pretreatment

After undergoing complete pretreatment (Step 4), samples S1, S2, M1, M2, and M3 exhibited a reduction in d_{50} values to the range of 6.83–12.54 μm compared to those subjected only to sieving. The particle size variation rates for these samples all exceeded 57%, with sample S1 showing the highest change rate of 73.23% (Table 1). This indicates that such sediments exist primarily as large aggregates under sieving-only treatment, leading to an overestimation of grain size in the measurements. In contrast, samples S3, N1, N2, and N3 showed minimal changes in d_{50} after complete pretreatment, with variation rates below 10%; sample S3 even exhibited a slight negative change (Fig. 3). This suggests that this group of samples may consist inherently of loosely structured coarse-grained sediments, or their particles possess chemically stable characteristics. The complete pretreatment process likely removed only the cementing matrix while effectively preserving the detrital framework dominated by quartz and feldspar.

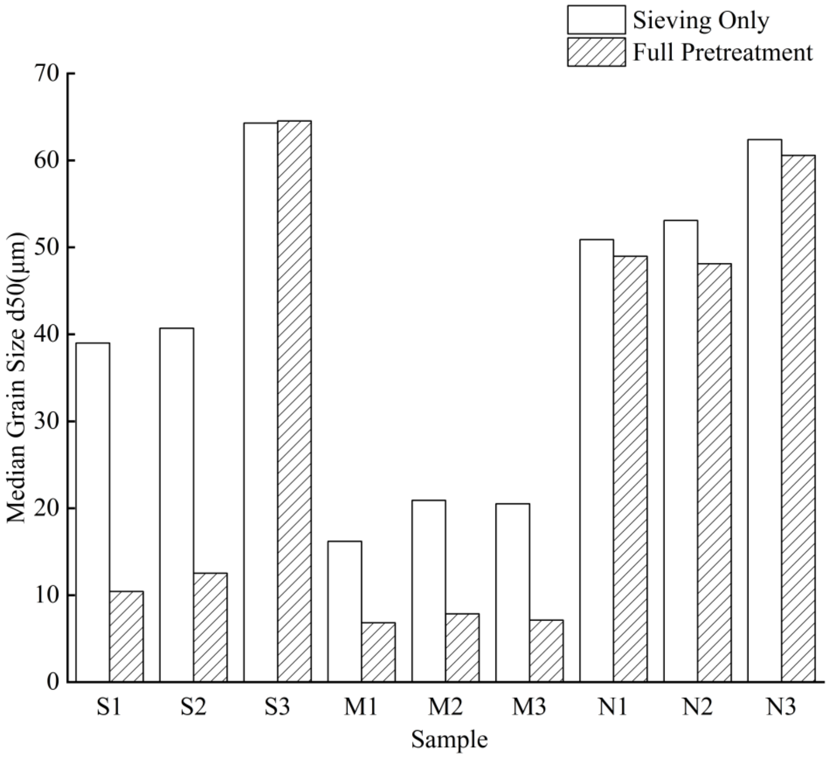

Fig. 3. Comparison of median grain sizes of desert river sediments across three sampling sections under different pretreatment conditions.

Table 1. Comparison of median grain sizes and their variation rates in desert river sediment samples between sieve-only treatment and complete pretreatment conditions.

Sample	dx_{50} Sieving only/μm	dx_{50} Complete pretreatment/μm	Variation Rate (%)
S1	39.00	10.44	73.23%
S2	40.70	12.54	69.19%
S3	64.30	64.54	-0.37%
M1	16.20	6.83	57.84%
M2	20.90	7.85	62.44%
M3	20.50	7.13	65.20%
N1	50.9	48.98	3.77%
N2	53.1	48.12	9.38%
N3	62.4	60.58	2.92%

Samples S2, M2, and M3 exhibit broad, bimodal or multimodal distribution curves when subjected only to sieving (Fig. 4), with the dominant peak skewed toward the coarser grain-size fraction. After undergoing complete pretreatment, the curve morphology changes significantly: the peak in the coarse fraction disappears, and the entire curve shifts markedly toward the finer grain sizes, transforming into a tall, narrow, unimodal distribution. This confirms that the acid-washing and oxidation steps effectively disaggregated coarse particle aggregates formed by silt and clay cementation.

For samples S3, N1, N2, and N3, the dominant peak morphology remains largely unchanged between the sieving-only and complete pretreatment methods, with consistent peak shape and position, which aligns with the observed trends in median grain size. Notably, although sample S3 shows minimal change in median grain size, a secondary peak is present at 1000 μm under sieving-only conditions, which completely vanishes after full pretreatment. This indicates that even for coarse-grained samples with a stable skeletal framework, complete pretreatment can effectively remove non-detrital components such as carbonate nodules or biogenic debris.

In summary, applying a complete pretreatment protocol is essential for obtaining the true grain-size distribution of desert river sediments.

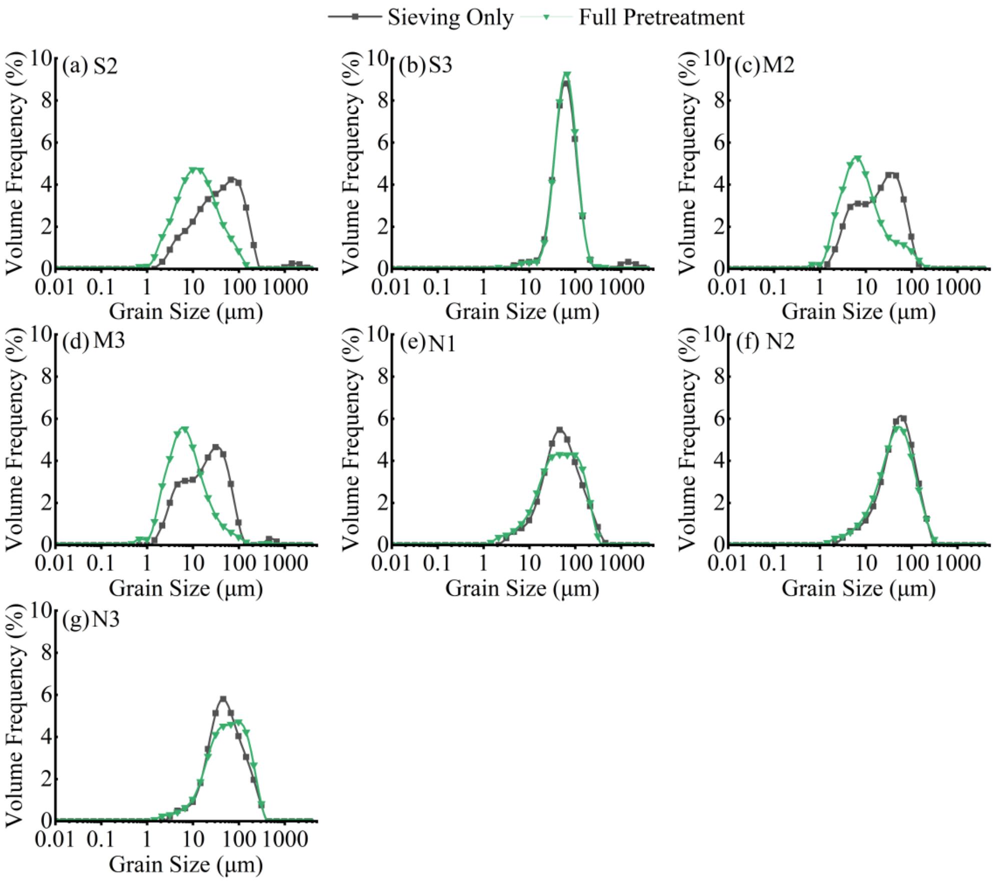

Fig. 4. Comparison of grain-size frequency distribution curves for desert river sediment samples under sieve-only treatment and complete pretreatment conditions.

4. Conclusion

In the terminal alluvial deposits of the Keriya River in the Taklamakan Desert, simply sieving river sediments prior to laser diffraction analysis is insufficient for accurately characterizing the grain size distribution of mineral particles. When applying different pretreatment procedures, it is essential to consider the specific characteristics of the samples and the influence of each pretreatment step on the final measurement outcomes. Carbonates are the predominant cementing agents in the study area, frequently encapsulating or binding fine-grained fractions into larger aggregates. Physical dispersion methods are primarily effective for breaking down loosely structured macro-aggregates; in contrast, acid digestion plays a decisive role in disaggregating tightly cemented micro-aggregates and liberating the finer components. The removal of organic matter mainly serves to eliminate interference caused by organic cementation and water-induced swelling in certain samples.

Acknowledgments

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ВЛИЯНИЕ ПРЕДВАРИТЕЛЬНОЙ ОБРАБОТКИ НА ЛАЗЕРНЫЙ ДИФРАКЦИОННЫЙ АНАЛИЗ РАЗМЕРОВ ЧАСТИЦ РЕЧНЫХ ОТЛОЖЕНИЙ В ПУСТЫННЫХ РЕГИОНАХ

Мэнменг Тан[1,2,3], Кери Яо[1,2,3], Цзин[1] Мэн[1,2,3], Ханьсяо Го[1,2,3], Хаоран Юань[1,2,3], Чжуньи Ху[1,2,3], Абдурейим Анвар[2,3,4], Юэ Дай[1,2,3]*, Фэн Чжан[1,2,3]*

[1]Колледж географии и дистанционного зондирования земли, Синьцзянский университет, Урумчи 830046, Китай;

[2] Хинцзянская полевая научно-исследовательская станция наблюдения за процессом оазисизации во внутренних районах пустыни Такла-Макан, Ютянь, Синьцзян 848400, Китай;

[3]Синьцзянская ключевая лаборатория экологии оазиса, Урумчи, 830046, Китай;

[4]Колледж экологии и охраны окружающей среды, Синьцзянский университет, Урумчи, 830046, Китай;

* Авторы-корреспонденты e-mail: daiyue@xju.edu.cn; zhangfeng@xju.edu.cn

Аннотация: Выбор методов предварительной обработки при лазерном дифракционном анализе минералогического состава зерен речных отложений может существенно повлиять на результаты измерений, однако опубликованные данные по этому вопросу остаются ограниченными. В этом исследовании систематически изучается влияние четырех различных методов предварительной обработки — только просеивания, только физического диспергирования, только удаления органического вещества и комбинированного удаления карбоната и органического вещества — на измерение размера зерен с использованием типичных речных отложений из конечного оазиса реки Керия в центральной части бассейна Тарима, в пустыне Такла-Макан. Результаты показали, что образцы, обработанные только методом просеивания, как правило, дают завышенные средние размеры зерен, причем в некоторых случаях степень изменения размера частиц превышает 70%. Мы пришли к выводу, что карбонат выступает в качестве основного связующего вещества между частицами осадка в этом регионе, а кислотное разложение играет решающую роль в дезагрегации микроагрегатов и выделении мелкозернистых фракций. Физическое диспергирование в основном эффективно для разрушения слабо структурированных макроагрегатов. Кроме того, удаление органических веществ имеет важное значение для устранения органической цементации и сведения к минимуму влияния водопоглощения и набухания некоторых образцов.

Ключевые слова: гранулометрический анализ; методы предварительной обработки; речные отложения; пустыня Такла-Макан.

ШӨЛДІ АЙМАҚТАРДАҒЫ ӨЗЕН ШӨГІНДІЛЕРІНІҢ БӨЛШЕКТЕРІНІҢ МӨЛШЕРІН ЛАЗЕРЛІК ДИФРАКЦИЯЛЫҚ ТАЛДАУҒА АЛДЫН АЛА ӨҢДЕУДІҢ ӘСЕРІ

Мэнменг Тан[1,2,3], Кери Яо[1,2,3], Цзин[1] Мэн[1,2,3], Ханьсяо Го[1,2,3], Хаоран Юань[1,2,3], Чжуньи Ху[1,2,3], Абдурейим Анвар[2,3,4], Юэ Дай[1,2,3]*, Фэн Чжан[1,2,3]*
[1]География және Қашықтықтан Зондтау ғылымдары колледжі, Шыңжаң Университеті, Үрімші 830046, Қытай;
[2]Такламакан Шөлінің Ішкі Бөлігіндегі Оазисизация Процесіне арналған шыңжаң Далалық Ғылыми Бақылау Және Зерттеу Станциясы, Ютянь, Шыңжаң 848400, Қытай;
[3]Xinjiang Оазис Экологиясының Негізгі Зертханасы, Үрімші 830046, Қытай;
[4]Экология және Қоршаған Орта колледжі, Шыңжаң Университеті, Үрімші 830046, Қытай;
Корреспондент-авторлар e-mail: daiyue@xju.edu.cn; zhangfeng@xju.edu.cn

Аннотация: Флювиальды шөгінділердегі түйіршіктердің минералогиялық мөлшерінің құрамын лазерлік дифракциялық талдауда алдын ала өңдеу әдістерін таңдау өлшеу нәтижелеріне айтарлықтай әсер етуі мүмкін, дегенмен осы тақырып бойынша жарияланған деректер шектеулі болып қала береді. Бұл зерттеу алдын ала өңдеудің төрт түрлі әдісінің әсерін жүйелі түрде зерттейді тек елеу, тек физикалық дисперсия, тек органикалық заттарды жою және карбонат пен органикалық заттарды бірге жою—орталық Тарим Бассейніндегі Керия өзенінің терминалдық оазисінен типтік ағынды шөгінділерді пайдалана отырып, түйіршіктердің мөлшерін өлшеуге, Такламакан шөлі. Нәтижелер көрсеткендей, тек елеу әдісімен өңделген үлгілер астықтың шамадан тыс орташа мөлшерін алуға бейім, кейбір жағдайларда бөлшектердің мөлшерінің өзгеру жылдамдығы 70% - дан асады. Біз карбонат осы аймақтағы шөгінді бөлшектер арасындағы негізгі цементтеуші зат ретінде әрекет етеді және қышқылдың қорытылуы микро агрегаттардың ыдырауында және ұсақ түйіршікті фракциялардың бөлінуінде шешуші рөл атқарады деген қорытындыға келдік. Физикалық дисперсия негізінен еркін құрылымдалған макро агрегаттарды ыдыратуда тиімді. Сонымен қатар, органикалық заттарды кетіру органикалық цементтеуді жою және белгілі бір үлгілерде судың сіңуіне және ісінуіне кедергілерді азайту үшін өте маңызды.

Түйін сөздер: астық мөлшерін талдау; алдын ала өңдеу әдістері; флювиалды шөгінділер; Такламакан шөлі.