

# An Improved Method of Laser Particle Size Analysis and Its Application in Identification of Lacustrine Tempestite and Beach Bar: An Example from the Dongying Depression

Yuanfu Zhang<sup>\*1,2</sup>, Chenlin Hu<sup>1,2</sup>, Xingmou Wang<sup>3</sup>, Min Wang<sup>4</sup>, Zaixing Jiang<sup>1,2</sup>, Junjie Li<sup>5</sup>

1. School of Energy Resources, China University of Geosciences (Beijing), Beijing 100083, China

2. Institute of Earth Sciences, China University of Geosciences (Beijing), Beijing 100083, China

3. SINOPEC Shengli Geophysical Research Institute, Dongying 257022, China.

4. PetroChina Research Institute of Petroleum Exploration and Development, Beijing 100083, China

5. The First Oil Production plant, SINOPEC Henan Oilfield, Henan 473132, China

**ABSTRACT:** Grain size analysis is a common method in the study of sedimentology. For the consolidated sedimentary rocks, the traditional methods are rock slice observation and image analysis. In recent years, laser particle size analyzer is used widely in particle size analysis of sedimentary rock. Unlike the pretreatment of loose samples, the rock samples must be crushed, added acid to wipe out cement, and washed. However, in the step of washing, most of the fines component (less than 63 $\mu$ m) in the suspended state should be inevitably lost. It will significantly affect the accuracy of particle size analysis, especially for siltstone. This paper presents a siltstone sample pretreatment method which core step is washing acid by centrifuge. Compared with traditional decantation method, the results show that the median particle size reduced 33.2 $\mu$ m on average. Compared with the precipitation method which is commonly used for handling loose samples, the change of solid-liquid separation time is from 12h to 10min, while the average reduction of median particle size is about 15 $\mu$ m. The grain size value corresponded to the cumulative volume of 10%/90% reduced 2.5 $\mu$ m/20.3 $\mu$ m on average. The percentage of the clay component less than 2 $\mu$ m increased 2.88% on average. The fine particle (2-4 $\mu$ m) and silt component (4-63 $\mu$ m) increased 1.71% and 5.56% on average. Based on this method, two kinds of similar lacustrine siltstone were analyzed. They are tempestite and beach bar which are difficult to identify in the Lijin sub-depression, Dongying depression, Shengli oilfield, China. The final grain-size probability plot of tempestite is the type of "one saltation component and three suspension components". The content of suspension components can reach to 80%-90%. The beach bar is the type of "one saltation component and two suspension components". The content of suspension components can reach to 40%-45%. They both have the characteristics of high slope which means well sorting. But they can be distinguished based on the suspension sedimentary characteristics which were preserved by maximum degree in this kind of sample pretreatment method.

**KEY WORDS:** Siltstone, Grain size analysis, Sample pretreatment, Washing acid, Centrifuge.

## 0 INTRODUCTION

Grain size analysis is a very important technique in sedimentological research (Cheetham et al., 2008; Rubina et al., 2007), as particle size and distribution are linked to the evolution of sedimentary environments and changes in sediment sources (Goossens, 2007).

Particle tests of sediments can include methods such as sieving and sedimentation (e.g., Zhang et al., 2011; Cheetham et al., 2008; Wang and Zhao, 2002; Beuselinck et al., 1998; Konert

and Vandenberghe, 1997; Singer et al., 1988). The laser particle size analysis method, which is one of the most important optical technologies developed in the 1970s, offers high precision and good identification of clay-sized particles (Ding et al., 2005). It has become increasingly popular in particle size analyses of sediments (Blott and Kenneth, 2006).

Analyses of particle size in sedimentary rocks often employ slice observations and image analysis. In recent years, laser particle size analysis has begun to be applied to sedimentary rocks. In contrast to the pretreatment of loose sediment samples, consolidated rock samples must be crushed, adding acid to remove any cement, and subsequently washed. The residual can be tested in the same way as unconsolidated materials.

Siltstone is widely distributed within a variety of sedimentary facies, including sheet sands (Hu et al., 2015; Jiang and Liu, 2011), shallow lake sandbars (Cao et al., 2009) and storm

\*Corresponding author: zyf@cugb.edu.cn

© China University of Geosciences and Springer-Verlag Berlin Heidelberg 2015

Manuscript received Jun 12, 2016.

Manuscript accepted Nov. 10, 2016.

Zhang, Y. F., Hu, C. L., Wang, X. M., et al., 2016. An Improved Method of Laser Particle Size Analysis and Its Application in Identification of Lacustrine Tempestite and Beach bar: An Example from the Dongying Depression. *Journal of Earth Science*, doi: 10.1007/s12583-016-0930-1. <http://en.earth-science.net>

deposits (Wang et al., 2009a). The grain size analysis of siltstone is important both for the identification of different sedimentary facies and for the reconstruction of paleocurrents (Bianchi et al., 1999). Though they are likely siltstones, the hydrodynamic conditions are complexity and different. In previous studies, the pretreatment procedures for siltstone-claystone samples (People's Republic of China Oil and Gas Industry Standard, 2009) have mainly included rock crushing, cement removal, washing acid, sample drying, and testing. However, owing to the small grain size of siltstone (less than 63  $\mu\text{m}$  in diameter) (Blott and Kenneth, 2006), fine components are easily lost in the process of washing acid.

Different washing acid processes have been developed. The decantation method means repeating washing after cement has been removed (Wang et al., 2003). As there is little time for suspended material to settle in this process, particles with diameter less than 40  $\mu\text{m}$  may be lost (Bianchi et al., 1999). Therefore, the decantation method should be considered most effective for larger grain size sandstones.

The standing method means to keep the mixture of water and sample standing for 12 hours (Konert and Vandenberghe, 1997), 24 hours (Wang and Zhu, 2005), or 72 hours (Pang et al., 2013), after cement removal. Then the supernatant is removed with a dropper pipette (Zhang et al., 2009; Wang et al., 2007; Cheng et al., 2001). However, the particles which less than 5  $\mu\text{m}$  settle extremely slowly. Even the complete settling cannot be

guaranteed within an experiment due to the effects of convection, diffusion, and Brownian motion (Allen, 1984).

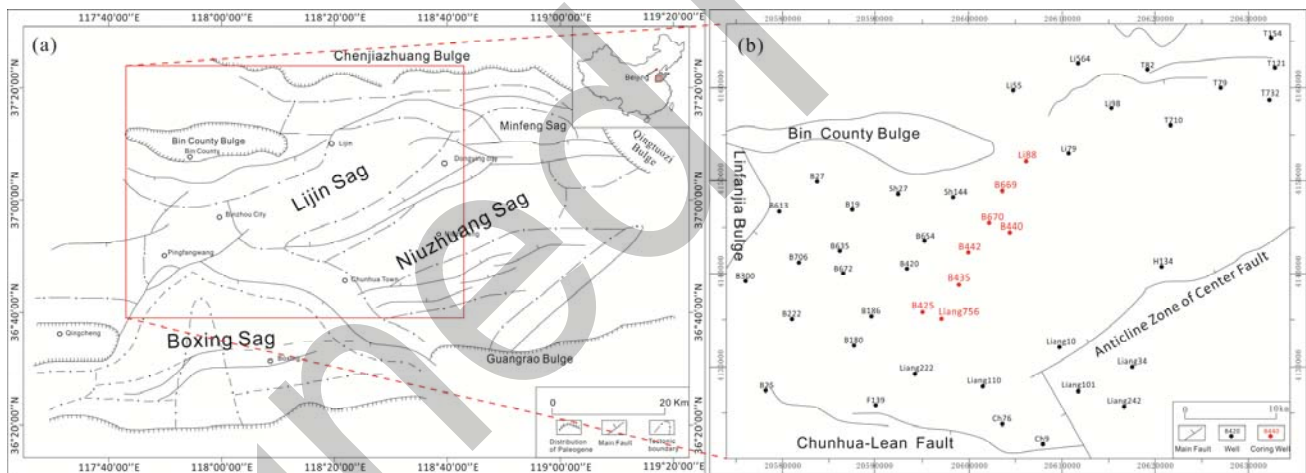
A set of pretreatment of laser grain size analysis for consolidated siltstone were studied in this paper. The aim is to get the entire information of grain size. Then the entire grain data should be applied to test if it is effective to identify likely siltstone such as lacustrine tempestite and beach-bar siltstone.

## 1 SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURE

The experimental procedure is as follows: 1) samples preparation of siltstone; 2) pretreatment with different methods; 3) grain size testing by Malvern Mastersizer 2000; 4) comparison and application of results.

### 1.1 Material preparation

Siltstone samples were taken from 25 cores, derived from several core wells (Table 1), including Bin425, Bin435, Bin 440, Bin 442, Bin 669, Bin 670, Liang 756, and Li 88, located in the Lijin sag, Dongying depression, in eastern China (Fig. 1). The cores belong to Paleogene Shahejie Formation which is 3000–4000 m underground. All the samples had been verified as lacustrine tempestite and beach-bar siltstone base on the typical sedimentary structures and the color of interbedding mudstone.



**Figure 1.** The wells are located in the Lijin Sag, Dongying Depression. The Lijin Sag is about 400km southeast of Beijing. (a) The north of the sag is adjacent to Chenjiazhuang Bulge. The west is Bin County Bulge. The southwest is Boxing Sag and the east are Minfeng Sag and Niuzhuang Sag. (b) The source of the samples are red highlight wells.

### 1.2 Sample pretreatment

(1) Rock crushing: Rock samples were crushed until the majority fragments diameter are less 1 mm. The entirety of the crushed sample must be transferred to a beaker.

(2) Organic matter clearance: 15%  $\text{H}_2\text{O}_2$  solution was poured into the sample beaker. Then the mixture was heated for 10 min. Alternatively, the beaker may be placed in a constant temperature water bath (50°C) to ensure a sufficient reaction between the hydrogen peroxide and organic matter.

(3) Cement elimination: 10% HCL solution was poured into the sample beaker after the removal of organic matter.

Calcareous cements may be considered fully removed when there was no further bubble generation.

(4) Washing acid: A centrifuge (TG18G- II type, it's RCF is 23950×g) was used to achieve the solid-liquid separation and retain all particle components. Firstly, the entire residue and mixture were transferred to a 100 ml centrifuge tube. Then deionized water was poured into the tube until the height of the water reached 6cm (2/3 of the tube). The speed of centrifugal separation was set to 11,000 rev/min. The sample should be centrifuged for 20 minutes. After that, most of the supernatant in the centrifuge tube was removed by a dropper pipette. The

process should be repeated until the pH of the supernatant is neutral. Finally, the residue was placed in a drying oven at 80°C for drying.

(5) Grinding: The dried, centrifuged residue was completely triturated into dispersed particles with an agate mortar.

(6) Particles dispersal: After grinding, the 0.5mol/L sodium hexametaphosphate solution was added to create a suspension.

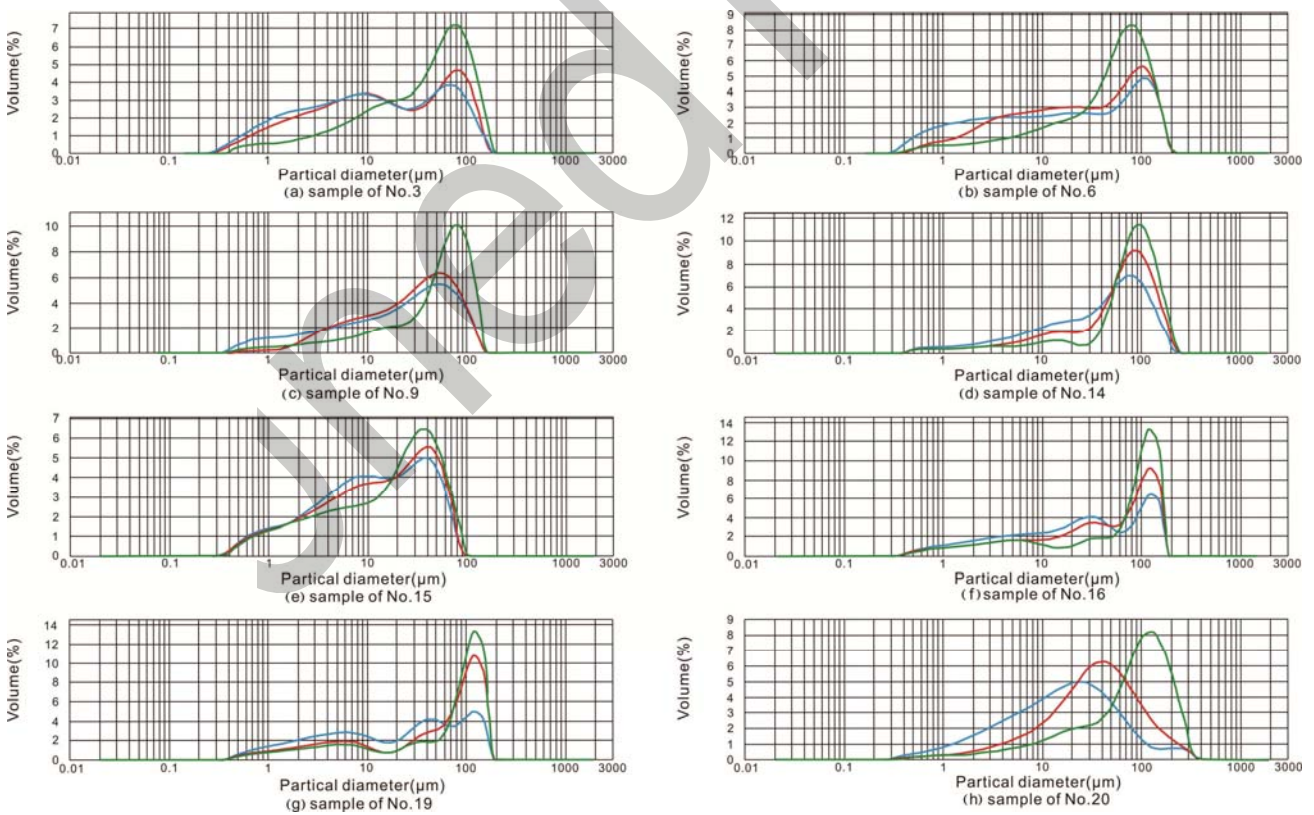
In the preparation process, thorough cleaning of each container guaranteed that no sample particles were lost. In addition,

high-speed centrifuge rotation ensured that extremely small particles were separated from the mixture according to Stokes' law in the centrifugal field. Thus, almost all components of siltstone sample would be retained and analyzed.

For comparison and verification, each sample was divided into nine parts after the step of rock crushing. Every three of them were used for centrifuge method, stand method (standing 12 hours) and decantation method (Fig.2, Table1).

**Table 1** The statistics of samples

Number	Well	Depth (m)	Maximum Diameter (cm)	Mass of Sample (g)	Number	Well	Depth (m)	Maximum Diameter (cm)	Mass of Sample (g)
1	B440	3845	9.6	78.6	14	B670	3480.7	10.3	82.3
2	B440	3850.7	9.8	79.8	15	Liang756	3067.5	10.4	83.1
3	B440	3921.2	9.7	79.1	16	Liang756	3070.1	9.8	79.6
4	B442	3895.8	9.6	78.7	17	Li88	3072.5	10.3	82.2
5	B442	3927.5	10.4	83.4	18	Li88	3076	10.8	87.6
6	B660	2759	10.6	85.6	19	Li672	4014.25	10.1	80.4
7	B660	3026	10.5	84.3	20	Li88	3090.8	10.4	83.9
8	B669	3486.4	10.3	82.1	21	Li88	3083	10.2	81.7
9	B669	3501.3	10.6	85.5	22	Li88	3069	10.7	86.4
10	B670	3272.8	10.2	81.6	23	Li88	3084	9.6	78.8
11	B670	3410.9	10.5	84.2	24	B442	3828.75	10.1	80.7
12	B670	3412.8	9.9	79.9	25	B669	3538.4	10.6	85.1
13	B670	3478.5	10.8	87.1					



**Figure 2.** Comparison of three pretreatment methods on samples No. 3, No. 6, No. 9, No. 14, No. 15, No. 16, No. 19, and No. 20. The red curve represents stand method, the blue curve represents centrifuge method, and the green curve represents decantation method. The results show that the volumes of the fine component (<63 μm) obtained from both the stand and centrifuge methods are larger than decantation methods.

### 1.3 Laser particle size analysis and data processing

Laser particle size analysis followed standard operating procedure of the Mastersizer 2000. It can provide the volume percentage, the distribution curve, and median size of particles. Reproducibility and contrast experiments were performed. Each sample was measured four times, and the most reproducible data were selected to ensure good reliability for comparison. The median grain size is summarized as Dv50. It corresponds to 50% of the cumulative volume. Accordingly, Dv10, and Dv90 are got. The percentage volume of each particles size fraction, including <2  $\mu\text{m}$ , 2–4  $\mu\text{m}$ , 4–63  $\mu\text{m}$ , and >63  $\mu\text{m}$ , were calculated based on cumulative results.

## 2 Results

### 2.1 Results comparison of the three pretreatment methods (Decantation, Standing and Centrifuge method)

Volume percentage distribution curves of each method were presented in one plot that shows the similarities of the curves (Fig. 2). Results clearly show that the stand and centrifuge methods are much more accurate than the decantation method. The volumes of the fine component (<63  $\mu\text{m}$ ) obtained from both the stand and centrifuge methods are increased, while the volume percentage of the coarse fraction volume is decreased. The volume of fine components obtained from decantation method is considerably reduced.

Furthermore, the volume percentage of the fine component obtained from centrifuge method is higher than that from the

stand method. This implies that the centrifuge method allows the retention of more fine components.

For all samples, several parameters of cumulative volume diameters show that the centrifuge method retains more fine components. The diameters corresponding to 10%, 50% and 90% of the cumulative volume are reduced by an average of 2.5  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 20.3  $\mu\text{m}$ , respectively, and Mz is decreased by 14.5  $\mu\text{m}$  (Table 2).

Table 3 also shows that the volume percentage of all clay-sized particle fractions, including <2  $\mu\text{m}$ , 2–4  $\mu\text{m}$ , and 4–63  $\mu\text{m}$ , are increased by an average of 2.9%, 1.7%, and 5.6%, respectively. The particles larger than 63  $\mu\text{m}$  are decreased by an average of 10.2%.

### 2.2 Lacustrine tempestite and beach-bar have different grain size characteristics.

Two typical kinds of siltstone were selected base on core observation in Lijin Sag, Dongying Depression. One was tempestite and the other was beach-bar.

The evidences of tempestite were hummocky bedding, "V" shaped tear shale cuttings, ruffled structure, gutter cast and other sedimentary structure which reflected stronger storm hydrodynamic conditions (Ma and Zhong, 1990; Liu, 1989). In addition, the adjacent dark gray shale also reflected the deep water (Fig. 3). On the other hand, low-angle cross-bedding, wavy bedding, inverse grain graded and gray-green mudstone were the features of shallow lake beach bar (Fig. 4).

**Table 2** Cumulative volume diameters for the stand method and the centrifuge method. The d10, d50 and d90 represent diameters corresponding to 10%, 50% and 90% of the cumulative volume, respectively, and Mz is mean grain size, given by  $(d_{16}+d_{50}+d_{84})/3$ . Units are all in micrometers.

Samples	Stand method ( $\mu\text{m}$ )				Centrifuge method ( $\mu\text{m}$ )			
	d10	d50	d90	Mz	d10	d50	d90	Mz
1	4.1	66.3	138.8	65.1	2.7	43.2	118.6	50.9
2	3.3	36.7	90.9	40.6	2.6	26.2	80.2	33.2
3	1.5	14.1	94.7	32	1.1	11.0	83.7	27.3
4	2.2	29.6	81.9	34.2	1.9	21.9	81.9	31.1
5	3.0	44.8	119.0	51.8	2.2	32.5	103.4	42.5
6	2.5	25.8	120.2	44.5	1.2	17.3	119.1	40.7
7	3.6	73.2	188.9	80.1	2.9	60.4	188.6	75.1
8	6.2	53.6	136.8	62.1	1.5	42.6	135.2	56.1
9	4.4	29.8	84.9	35.9	1.8	24.1	83.1	32.2
10	9.3	81.2	157.8	79.9	3.0	45.1	125.4	51.9
11	12.2	75.9	145.2	75.4	4.7	58.6	138.7	62.4
12	8.2	85.5	174.8	86.1	4.9	76.6	163.1	53.1
13	3.4	49.7	109.7	51.5	2.4	30.8	91.4	37.7
14	7.6	66.9	139.9	66.8	4.4	45.3	121.9	52.5
15	1.8	14.1	51.8	20.3	1.7	12.1	49.1	18.5
16	2.8	48.7	139.3	60	2.0	26.3	130.6	49.3
17	20.1	100.3	187.8	104.5	6.8	86.9	170.9	81.8
18	1.8	18.2	59.4	23.3	1.6	14.5	51.0	19.3
19	2.4	68.8	142.9	67.7	1.6	23.3	119.4	40.9
20	6.3	34.7	115.9	45.2	2.3	16.6	70.7	24.2
21	1.3	14.4	51.5	19.4	1.1	9.7	42.0	15.3
22	5.6	51.3	243.7	85.4	4.5	39.1	186.6	61.9
23	7.9	68.3	346.9	125.1	5.1	46.5	254.1	87.3
24	7.8	43.2	253.5	87.4	4.7	30.6	163.5	47.0
25	8.9	63.0	238.4	90.9	7.7	41.7	235.0	78.9



**Figure 3.** Cores of tempestite. (a): Well Bin 666, 3094.1m, "V" shaped tear shale cuttings; (b): Bin 440, 3848.15m, rumbled structure; (c): Bin 440, 3842.65m, dark gray mudstone; (d): Li672, 4132.5m, gutter cast; (e): Bin 670, 3282m, hummocky bedding; (f): Bin 440, 3918.3m, mix structure.

By means of Centrifuge method and laser grain analyses, the fine component ( $<63 \mu\text{m}$ ) of tempestite and beach bar were increased. As a result, some difference can be found in the cumulative-probability plots.

Well Bin669 and Liang756 represent tempestite samples. The range of grain size is from  $2\phi$  to  $12\phi$  ( $250\mu\text{m}$ - $0.2\mu\text{m}$ ). The cumulative probability plots can be divided into four parts generally. According the grain size analyses method, the lowest part is the "saltation subfabric", the other parts are "suspension subfabric". The junction point between saltation and suspension parts is about  $3.5-4\phi$  ( $88.4\mu\text{m}$ - $62.5\mu\text{m}$ ). The suspension content is close to 80-90% (Figs. 5a and 5b).

Well Bin440 and Bin670 represent beach-bar samples. The range of grain size is from  $2\phi$  to  $12\phi$  ( $250\mu\text{m}$ - $0.2\mu\text{m}$ ). The cumulative probability plots can be divided into three parts generally. The lowest part is the "saltation subfabric". The junction point between saltation and suspension parts is about  $4\phi$  ( $62.5\mu\text{m}$ ). The saltation content is close to 55-60% (Figs. 5c and 5d).

### 3 DISCUSSION

#### 3.1 Influence of centrifugation time and speed

The Stokes' equations in the centrifugal field was summarized as:

$$\frac{dx}{dt} = u_c = \frac{(\rho_s - \rho_f)}{18\eta} D^2 \omega^2 x \quad (\text{Allen, 1984})$$

$x$  -the distance between particles and axis of rotation;

$\frac{dx}{dt}$  -the outward velocity of particles;

$\rho_s$  -density of the particle;

$\rho_f$  -density of the medium;

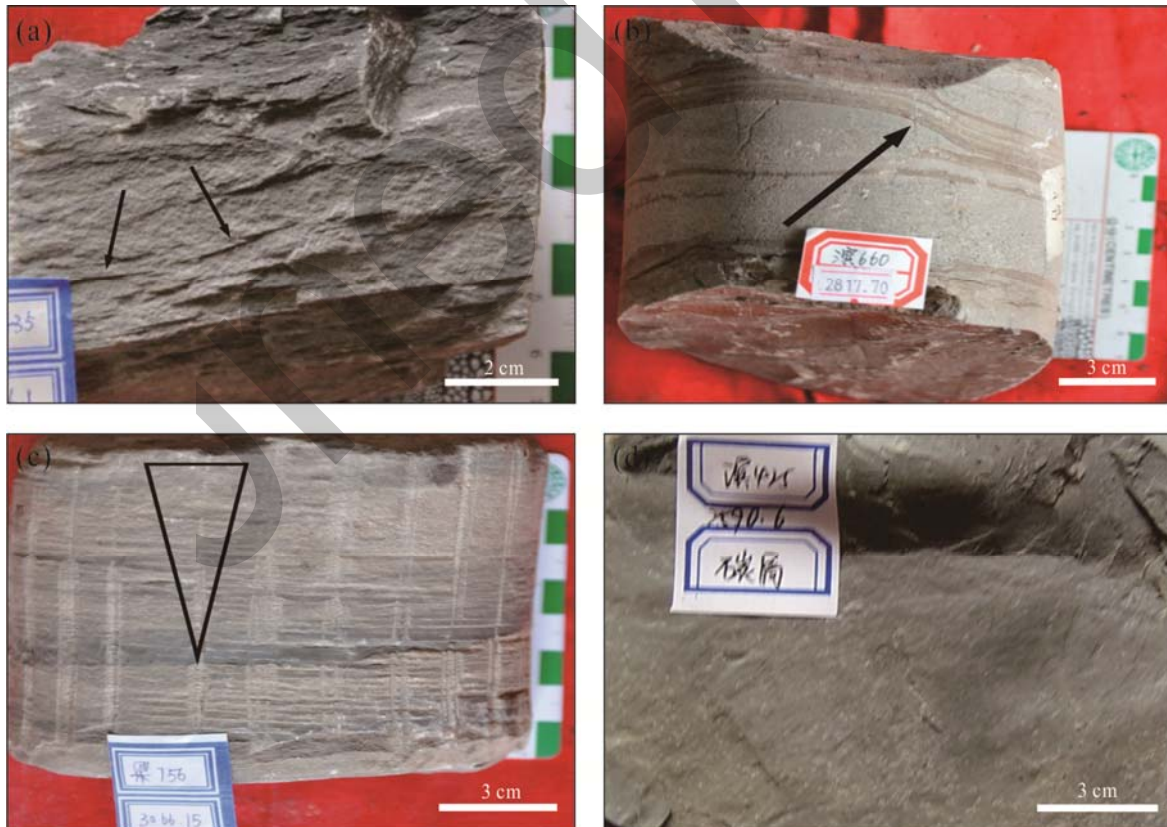
$\eta$  -viscosity in poises;

$D$  -diameter of the particle;

$\omega$  -the angular velocity of the centrifuge.

**Table 3** Percentage of different particle size fractions from the stand method and centrifuge method

Samples	Stand method				Centrifuge method			
	<2 $\mu$ m	2-4 $\mu$ m	4-63 $\mu$ m	>63 $\mu$ m	<2 $\mu$ m	2-4 $\mu$ m	4-63 $\mu$ m	>63 $\mu$ m
1	5.34%	4.53%	38.34%	51.79%	7.51%	6.33%	50.76%	35.4%
2	6.55%	4.99%	62.29%	26.17%	8.05%	6.71%	66.96%	18.28%
3	14.1%	10.41%	53.86%	21.63%	18.38%	11.75%	53.02%	16.85%
4	9.4%	7.02%	62.69%	20.89%	10.83%	8.06%	64.21%	16.9%
5	7.1%	5.41%	50.4%	37.09%	9.03%	7.6%	54.5%	28.87%
6	7.65%	8.9%	52.06%	31.39%	17.32%	10.03%	44.92%	27.73%
7	6.45%	4.24%	35.38%	53.93%	7.37%	4.83%	39.23%	48.57%
8	4.58%	2.55%	47.44%	45.43%	11.5%	3.3%	44.7%	40.5%
9	2.42%	6.29%	71.24%	20.05%	11.1%	6.44%	64.39%	18.07%
10	2.9%	2.61%	30.91%	63.58%	6.52%	6.8%	47.46%	39.22%
11	2.56%	2.02%	34.52%	60.9%	4.47%	4.13%	44.53%	46.87%
12	3.09%	2.83%	31.66%	62.42%	4.29%	3.97%	35.24%	56.5%
13	6.21%	5.3%	49.4%	39.09%	8.53%	7.2%	60.77%	23.5%
14	3.3%	2.67%	41.02%	53.01%	4.93%	4.04%	53.25%	37.78%
15	12.21%	10.12%	73.12%	4.55%	11.8%	11.47%	75.64%	1.09%
16	7.4%	5.8%	42.75%	44.05%	10.21%	7.94%	51.49%	30.36%
17	1.68%	1.43%	21.3%	75.59%	4.17%	3.14%	26.94%	65.75%
18	10.41%	9.57%	70.78%	9.24%	12.88%	9.95%	72.62%	4.55%
19	8.54%	6.71%	32.51%	52.24%	12.81%	10.16%	50.54%	26.49%
20	3.1%	3.35%	67.38%	26.17%	8.51%	8.89%	70.7%	11.9%
21	16.33%	10.41%	67.02%	6.24%	19.29%	12.86%	65.65%	2.2%
22	4.01%	3.54%	48.11%	44.34%	4.79%	4.34%	55.92%	34.95%
23	2.95%	2.77%	42.61%	51.67%	4.28%	3.9%	50.09%	41.73%
24	3.11%	2.75%	55.85%	38.29%	4.06%	4.54%	65.92%	25.48%
25	2.33%	2.28%	45.28%	50.11%	3.12%	2.78%	57.53%	36.57%



**Figure 4.** Cores of beach-bar. (a): Well Bin 435, 3346.1m, low-angle cross-bedding; (b): Bin 660, 2817.7m, wavy bedding; (c): Liang756, 3366.15m, inverse grain graded; (d): Bin425, 2590.6m, gray-green mudstone.

These parameters were measured in the experiment. The  $x$  was 5cm,  $\rho_s$  was  $2.6 \times 10^3 \text{ kg/m}^3$  (average of these samples),  $\rho_f$  was  $0.998 \times 10^3 \text{ kg/m}^3$  ( $20^\circ\text{C}$ ),  $\eta$  was  $1 \times 10^{-3} \text{ Pa}\cdot\text{s}$  ( $20^\circ\text{C}$ ),  $D$  was  $0.1\mu\text{m}$ , and the  $n$  was 11000 rev/min.  $0.1\mu\text{m}$  is the boundary of colloid and suspension. The colloidal particles between 1nm-100nm do not interact with the hydrodynamic conditions, so they could be ignored in the study of sedimentary rocks.

Then All parameters were brought into the equation. The distance ( $x$ ) that the particle moved in 20 minutes was calculated to be 7cm. It was greater than the length of water in the centrifuge tube. So almost all the particles ( $>0.1\mu\text{m}$ ) could be separated in the centrifuge tube.

### 3.2 The influence of acid on the clay minerals

In the grain size analysis of the mudstones (Jiang and Liu, 2011) or soil (Pang et al., 2013), the operation of adding acid were mentioned in the pretreatment process.

In this experiment, the acid with different concentration were used in pretreatment process. The influence of the acid on the clay was analyzed by the laser particle size analysis results.

From the table , adopting different concentrations of acid (5%-30%) in 20min, the results of Dv10, Dv50 and Dv90 were similar. Especially, the Dv10, representing the clay component, changed only in  $0.1\mu\text{m}$ . And there was a little change for the volume of the particles less than  $2\mu\text{m}$ . The range of change is about  $\pm 0.5\%$ . Moreover, if the pretreatment time was extended to 2h with 30% HCL, the volume of the particles less than  $2\mu\text{m}$

would increase (Table 4). Compared with 20 minutes experiment, the range of change was about  $\pm 1.2\%$ . In addition, the clay minerals have no obvious sensitivity to acid except montmorillonite. In the exchange of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^3$ ,  $\text{Fe}^{2+}$  with  $\text{H}^+$ , bond breaking is essential and the reaction is slow. As a result, if time is less than 20min and HCL is 5% and 10%, it is clearly that the acid has little effect on the results.

### 3.3 Applicability of the laser grain size analysis with centrifuge

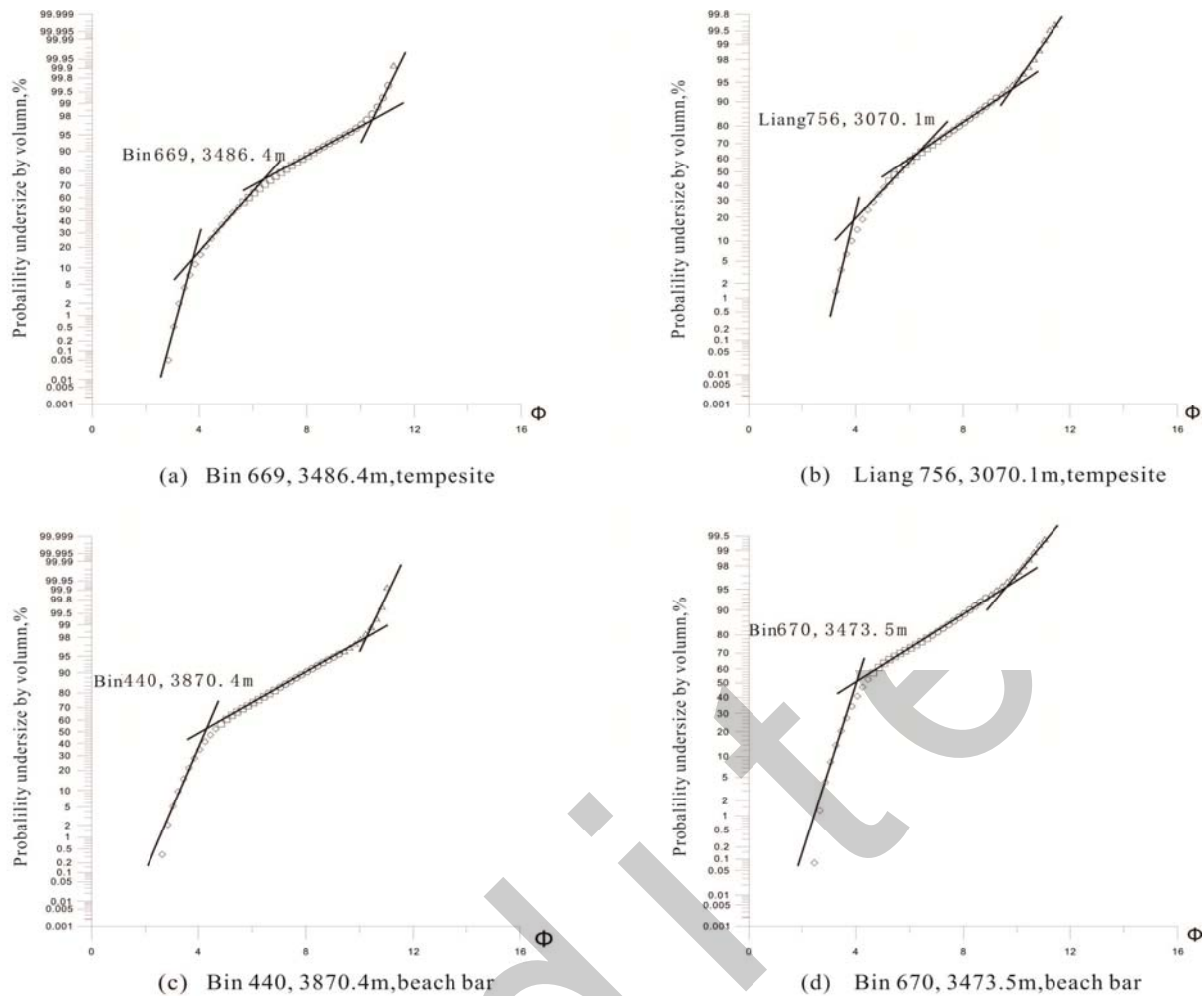
The laser grain size analysis with centrifuge has many advantages. It is fast, effective, ease of repetition, and ease of application to large numbers of samples. It makes it possible to identify two similar siltstone, beach-bars and storm deposits.

Though some scholars had summarized the characteristics of the two similar siltstones (Cao et al., 2009; Wang et al., 2009a,b; Yuan et al., 2005; Yuan et al., 2003), the grain size differences between them were not indicated or explained.

Firstly, they have different number of subfabric. The different subfabric reflects different hydrodynamic conditions (Jiang, 2010). Secondly, the proportions of the bouncing and the suspension subfabric are different. The storm deposits were dominated by suspension subpopulation and the proportion is about 80%-90%. The higher slope of each subpopulation showed good sorting of the siltstones. On the contrary, the beach-bar siltstones are dominated by the bouncing subpopulation and the proportion is about 55%-60%, the proportion of suspensions is less than 40%.

**Table 4** The influence of the acid on the laser grain size analysis

Acid Concentration	Grain Size	Sample Number							
		1	The Volume of the Particles $<2\mu\text{m}$	2	The Volume of the Particles $<2\mu\text{m}$	3	The Volume of the Particles $<2\mu\text{m}$	4	The Volume of the Particles $<2\mu\text{m}$
5%	Dv10	1.981		1.713		1.674		1.289	
	Dv50	28.678	9.7%	23.254	11.0%	22.937	11.2%	13.641	14.6%
	Dv90	116.679		68.144		79.211		88.367	
10%	Dv10	1.952		1.706		1.688		1.286	
	Dv50	28.563	9.8%	23.077	11.0%	22.972	11.2%	13.060	14.7%
	Dv90	116.004		67.501		76.848		77.107	
20%	Dv10	1.927		1.687		1.666		1.276	
	Dv50	28.405	9.9%	22.681	11.2%	22.317	11.2%	13.366	14.8%
	Dv90	115.839		67.379		74.115		78.530	
30%	Dv10	1.885		1.680		1.624		1.253	
	Dv50	28.173	10.1%	22.405	11.2%	22.132	11.5%	13.143	15.1%
	Dv90	115.543		67.280		73.959		78.419	
30%(2 hours)	Dv10	1.809		1.551		1.538		1.177	
	Dv50	27.678	10.5%	21.686	12.2%	21.647	12.1%	12.430	16.3%
	Dv90	114.917		66.890		73.546		76.435	



**Figure 5.** grain cumulative-probability plots of tempestitute and beach-bar. (a) and (b): Well Bin669 and Liang756 represent tempestitute samples. They have “one saltation component and three suspension components”. The content of suspension components can reach to 80%-90%. (c) and (d): Well Bin440 and Bin670 represent beach-bar samples. They have “one saltation component and two suspension components”. The saltation content is close to 55-60%.

#### 4 CONCLUSION

This paper presents and describes the centrifuge method as a complete procedure for siltstone sample pretreatment. By using a centrifuge in washing acid, more fine components are retained by this new method than the traditional decantation and stand methods. Compared with traditional decantation technique, this new method retains more fine components, and the median particle size of samples is reduced by an average of 33.2  $\mu\text{m}$ . Further, compared with the stand method, the solid-liquid separation time decreases from at least 12 hours to 20 minutes.

The centrifugation has two main advantages: 1) it is rapid and has good reproducibility, so it is suitable for dealing with a large number of samples; 2) the maximum number of fine components are retained from siltstone sample, ensuring that nearly all components are accurately detected.

The fine components (<63  $\mu\text{m}$ ) directly correspond hydrodynamic conditions for siltstones and muddy siltstones deposited in a lake or ocean. It is useful for researching depositional environment and original hydrodynamic conditions.

This method provides a new idea to distinguish the shallow lake sandbars and the storm deposits, particularly for the siltstones without clear sedimentary structures. The final grain-size probability plot of tempestitute is the type of “one saltation component and three suspension components”. The content of suspension components can reach to 80%-90%. The beach bar is the type of “one saltation component and two suspension components”. The content of suspension components can reach to 40%-45%.

#### ACKNOWLEDGMENTS

This work was supported by the National Natural Science Foundation of China (41572092), and China Key Laboratory of Land and Resources Ministry of shale gas resource evaluation and Strategy selection. The laboratory studies discussed in this paper were carried out at the Laboratory Center of School of Energy Resources, China University of Geosciences (Beijing). Thanks are also extended to the anonymous reviewers for their constructive comments.



## REFERENCES CITED

- Allen, T., 1984. Particle size measurement(3Ed). China Building Industry Press, Beijing, 219-414.
- Beuselink, L., Govers, G., Poesen, J., et al., 1998. Grain-size analysis by laser diffractometry: comparison with the sieve-pipette method. *Catena*, 32(3):193-208
- Bianchi, Hall., McCave., et al., 1999. Measurement of the sortable silt current speed proxy using the sedigraph 5100 and coulter Multisizer Iie: precision and accuracy. *Sedimentology*, 46(6): 1001-1014
- Blott, S. J., Kenneth, P., 2006. Particle size distribution analysis of sand-sized particles by laser diffraction: an experimental investigation of instrument sensitivity and the effects of particle shape. *Sedimentology*, 53(3): 671-685
- Cao, Y. C., Wang, J., Liu, H. M., et al., 2009. Sedimentary characteristics and models of beach-bar sandbodies in the upper part of the fourth member of Paleogene in the south slope of Dongying depression. *Journal of China University of Petroleum*, 33(6): 5-10
- Cheetham, M. D., Keene, A. F., Bush, R. T., et al., 2008. A comparison of grain-size analysis methods for sand-dominated fluvial sediments. *Sedimentology*, 55(6): 1905-1913
- Cheng, P., Gao, S., Li, X. S., 2001. Evaluation of a wide range laser particle size analysis and comparison with pipette and sieving methods. *Acta sedimentologica sinica*, 19(3): 449-454 (in Chinese with English abstract)
- Ding, X. G., Ye, S. Y., Gao, Z. J., 2005. Development and application of grain size analysis technique. *Global Geology*, 24(2): 203-207 (in Chinese with English abstract)
- Goossens, D., 2008. Techniques to measure grain-size distributions of loamy sediments: a comparative study of ten instruments for wet analysis. *Sedimentology*, 55(1): 65-96
- Hu, C.L., Zhang, Y. F., Jiang, Z. X., et al., 2015. Morphologic Changes in Modern Onshore Beach Bar of Poyang Lake under Wind and Wave Actions. *Acta Petrolei Sinica*, 36(12):1543-1552
- Jiang, Z. X., 2010. *Sedimentology*. Petroleum Industry Press, Beijing, China, pp. 159-161 (in Chinese)
- Jiang, Z. X., Liu, L. A., 2011. A pretreatment method for grain size analysis of red mudstones. *Sedimentary Geology*, 241(1-4): 13-21
- Konert, M., Vandenberghe, J., 1997. Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44(3): 523-535
- Liu, S. C., 1989. The characteristics of tempestite and the different sign contrasting with turbidites. *Sedimentary facies and Palaeogeography*, 05: 44-55+43 (in Chinese)
- Ma, Y. S., Zhong, L., 1990. Recent development of study on storm deposition and tempestites. *Geological Science and Technology Information*, 9(3): 9-14 (in Chinese with English abstract)
- Pang, J. L., Qiao Jing., Huang, C. C., 2013. Pretreatment methods and their influences on grain-size measurement of palaeosol in the upper reaches of the Hanjiang River Valley, China. *Scientia geographica sinica*, 33(6): 748-754 (in Chinese with English abstract)
- People's Republic of China Oil and Gas Industry Standard (2009) SY/T 5434-2009: Analysis method for particle size of clastic rocks. People's Republic of China Oil and Gas Industry Standard 14 pp. National Energy Bureau.
- Rubina, D. M., Chezarb, H., Hamey, J. N., et al., 2007. Underwater microscope for measuring spatial and temporal changes in bed-sediment grain size. *Sedimentary Geology*, 202(3): 402-408
- Singer, J. K., Anderson, J. B., Ledbetter, M. T., et al., 1988. An assessment of analytical techniques for the size analysis of fine-grained sediments. *Journal of Sedimentary Research*, 58(3): 534-543
- Wang, D. J., Fan, D. D., Li, C. X., 2003. Influence of different pretreatment on size analysis and its implication. *Journal of Tong Ji university*, 31(3): 314-318 (in Chinese with English abstract)
- Wang, J. B., Zhu, L. P., 2005. Influence of different Pre-treatments on grain-size measurement of lake sediments. *J. Lake Sci*, 17(1): 17-23 (in Chinese with English abstract)
- Wang, J. B., Ju, Y. T., Zhu, L. P., 2007. Comparison of lake sediment grain size results measured by two laser diffraction particle size analyzers. *J. Lake Sci*, 19(5): 509-515 (in Chinese with English abstract)
- Wang, S. L., Jiang, Z. X., Liu, H., 2009a. Sedimentary characteristics and model of tempestites-beach bar of upper section of the fourth member of Shahejie Formation in Boxing Sag. *Fault-Block oil & gas filed*, 16(4): 37-39 (in Chinese with English abstract)
- Wang, X. G., Zhao, B. H., 2002. Measurement technique and application of power particle size. *China molybdenum industry*, 26(6): 32-34 (in Chinese with English abstract)
- Wang, Y. M., WANG, X. H., Gao, Y. S., 2009b. Modern methods for the measurement and characterization of particle size in geosdandards reference materials. *Geological Bulletin of China*, 28(1): 137-145 (in Chinese with English abstract)
- Yuan, J., Du, Y. M., Li, Y. N., 2003. Probability cumulative grain size curves intertrogenous depositional environments of the Paleogene in Huimin Sag. *Petroleum Exploration and Development*, 30(3): 103-106 (in Chinese with English abstract)
- Yuan, W. F., Chen, S. Y., Zeng, C. M., 2005. Probability cumulative grain size curves in terrigenous of the Tertiary in west Qaidam Basin. *Journal of the University of Petroleum*, 29(5): 12-18 (in Chinese with English abstract)
- Zhang, Y. F., Jiang, Z. X., Wang, Y., et al., 2009. Comparative Analysis of Sequence Characteristics among Different Superimposed Stages of the Chelif Basin, Algeria. *Acta Geologica Sinica (English Edition)*, 83(6): 1041-1051
- Zhang, Y. F., Jiang, Z. X., 2011. Outcrop characterization of an early Miocene slope fan system, Chelif Basin, Algeria. *Energy Exploration and Exploitation*, 29(5): 633-646