

Particle Characterisation

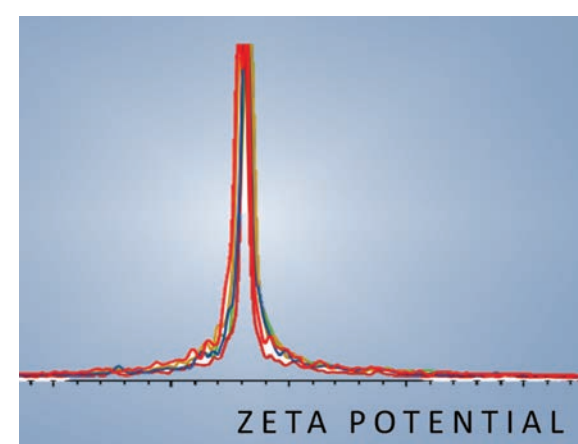
Faster, More Sensitive Zeta-Potential Measurements with cmPALS and the Litesizer™ 500

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Zeta potential is a key indicator of colloid stability. The higher the magnitude of the zeta potential (that is, highly positive or highly negative), the more stable the colloid. A lower-magnitude zeta potential indicates a less stable colloid; in other words, the colloidal particles will tend to aggregate or coagulate. Thus, knowledge of zeta potential is important for optimising processes and for quality control.

Until now, the state of the art in zeta-potential measurements was phase-analysis light scattering (PALS) [1], which is based on electrophoretic light scattering (ELS) measurements. PALS was an improvement over previous ELS measurements because it no longer required the application of such large fields, so sample heating and decomposition was reduced. PALS measurements can be unreliable, however, especially with sensitive samples, where very short measuring times and low electric fields are required.

We describe herein a newly patented technology called cmPALS (EP2735870) [2], which is incorporated in the recently introduced particle-analysing instrument, the Litesizer™ 500. cmPALS results in dramatic enhancements in the sensitivity and stability of zeta-potential measurements. We describe first the technique of cmPALS before presenting a direct comparison of results from the cmPALS instrument (the Litesizer™ 500) with those from a PALS instrument.



Continuously Monitored Phase-Analysis Light Scattering (cmPALS)

Our research team at Anton Paar have developed a modification of PALS in which an additional modulator monitor is implemented (see Figure 1). In the newly patented method, “continuously monitored PALS” (cmPALS), this extra modulator monitor detects the interference between the modulated and un-modulated (reference beam) laser light (Figure 1, Figure 2). Thus, its beat frequency is solely the modulation frequency and is therefore independent of the electrophoretic motion of the particles. In other words, the frequency difference between sample interference and modulator monitor is exactly the Doppler shift caused by the electrophoretic motion of the particles. Any non-linearity of the modulator is automatically compensated, and does not influence the results. The quality of the results is also not affected by any longer-term deterioration in the modulator performance. Thus, the advent of cmPALS means that modulators with large movements can be used, despite their non-linearities. As a consequence the sensitivity and stability of laser Doppler electrophoresis measurements can be significantly enhanced because measurements of substantially shorter duration and lower electric field applied become feasible.

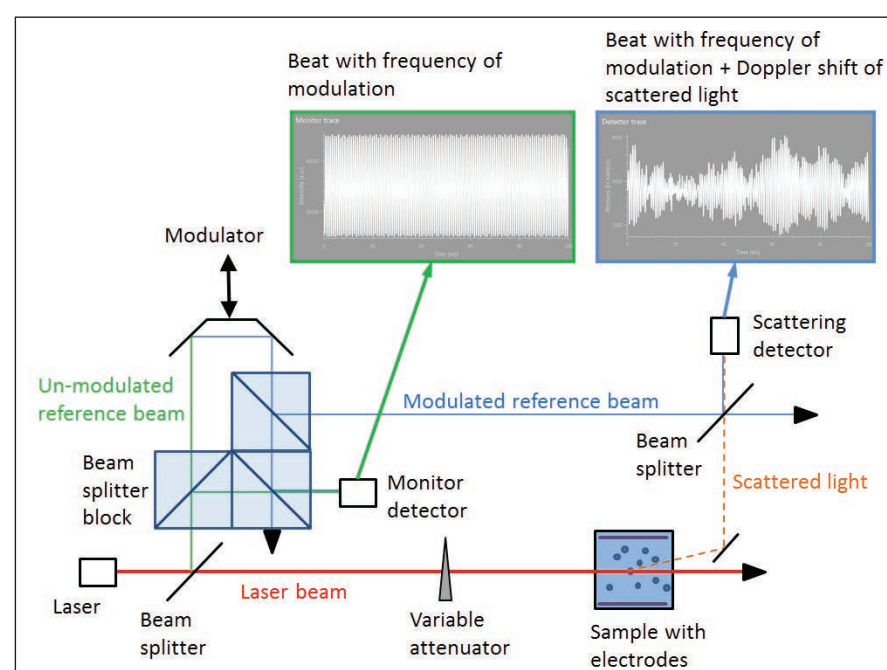


Figure 1. Schematic representation of cmPALS as implemented in the Litesizer™ 500.

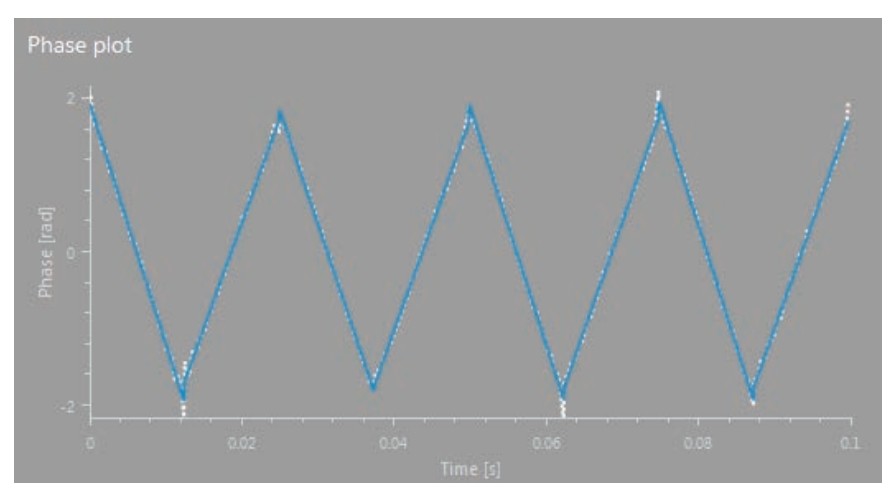


Figure 2. Typical phase plot of an ELS measurement with cmPALS data analysis.

Experimental

Latex standard solution was purchased from Thermo Fisher Scientific and diluted to 0.05% in 10 mM NaCl. Zeta potential measurements were carried out with cmPALS technology on a Litesizer™ 500 particle analyser, and with PALS technology on a competitor's instrument.

Results and Discussion

To compare the performance of cmPALS and PALS, the zeta potential of a latex standard (0.05% in 10 mM NaCl) was measured with a cmPALS instrument and a PALS instrument (Table 1).

Zeta potential was monitored as the number of runs, and hence measurement time, decreased. The results show that cmPALS produces results with increased precision in a substantially lower measurement time than that required by the PALS instrument. In fact, when the number of runs is reduced such that the measurement time is only 2 seconds, the Litesizer™ 500 still provides high-quality results with very low error (standard deviation <1%).

The performance of cmPALS vs. PALS was also tested at low electric fields by measuring the zeta potential at decreasing voltages. Again, the sample was a latex standard (0.05% in 10 mM NaCl).

Although both methods show a decreasing zeta potential magnitude as the voltage is reduced (see Table 2), there is a significant difference in the quality of the results. At the lowest voltage, the Litesizer™ 500 measurements are still fast (11 s), and also show a low standard deviation. In contrast, measurements with the PALS device take much longer (31 s to 70 s) and the standard deviation remains higher. Thus, the cmPALS measurements show better repeatability, and are much faster than the PALS measurements.

Table 1: cmPALS vs PALS: Zeta potential measurements with varying measurement times.
Sample: latex standard (0.05 % in 10 mM NaCl). SD = standard deviation, RSD = relative standard deviation

cmPALS			PALS		
ZP [mV] ^a	SD [mV] (RSD)	Measurement duration [s]	ZP [mV] ^a	SD [mV] (RSD)	Measurement duration [s]
-61.2 ^b	±0.7 (1.1 %)	10	-57.9 ^b	±1.8 (3.1 %)	31
-61.0	±0.7 (1.1 %)	8	-57.3	±2.3 (4.0 %)	26
-61.3	±0.1 (0.2 %)	6	-59.7	±1.3 (2.2 %)	13
-60.9	±0.5 (0.8 %)	4	-57.1	±2.1 (3.7 %)	5
-60.9	±0.4 (0.7 %)	2	-58.1	±2.6 (4.5 %)	3
Mean: -61.0 ± 0.5			Mean: -58.0 ± 2.0		
RSD: 0.8%			RSD: 3.4%		

- a. Each result is the mean value of three consecutive measurements
b. Measurement performed in automatic mode

Table 2: cmPALS vs PALS: zeta-potential measurements at decreasing voltages.
Sample as for Table 1. SD = standard deviation, RSD = relative standard deviation

cmPALS				PALS			
ZP [mV] ^a	SD [mV] (RSD)	Voltage applied [V]	Measurement duration [s]	ZP [mV] ^a	SD [mV] (RSD)	Voltage applied [V]	Measurement duration [s]
-61.2 ^b	±0.7 (1.1 %)	200	10	-57.9 ^b	±1.8 (3.1 %)	148	31.2
-58.0	±0.5 (0.9 %)	100	10	-54.0	±1.0 (1.9 %)	100	31.2
-55.0	±0.3 (0.5 %)	50	14	-49.0	±1.6 (3.3 %)	50	36.4
-55.0	±1.2 (2.2 %)	20	11	-46.4	±3.0 (6.5 %)	20	70.2

- a. Each result is the mean value of three consecutive measurements
b. Measurement performed in automatic mode

Summary

Continuously monitored phase-analysis light scattering (cmPALS) is a development of the classical PALS method. The result is that the sensitivity and stability of laser Doppler electrophoresis measurements are dramatically enhanced. For the user, this means significantly shorter measurement times and lower electric fields, and therefore more sensitive samples can be analysed with confidence.

References

1. W. Tscharnuter, *Mobility measurements by phase analysis*, *Applied Optics* 2001, 40:24, 3995-4003.
2. H. Noack; C. Moitz, *Modulator monitoring during measuring electromobility*. European Patent EP2735870, April 25th, 2015.

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