

Particle concentration, size and turbidity

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The amount of light scattered by particles in a suspension depends on the concentration and geometry of the particles, and on the optical properties of the suspension. In the paper a new method is presented to extract particle concentration and a characteristic particle size from turbidimeter readings. The method requires a turbidimeter capable of measuring the forward (12°) and sideways (90°) scattered light simultaneously. The turbidimeter must be calibrated with several samples with known particle concentration and characteristic size. Results are presented for the calibration of filter aids, an industrial pigment and yeast. An example is given of its intended use: the on-line analysis of filtrate turbidity. The method is of course limited to the range of particle concentration (1–200 ppm) and characteristic particle size (1–100 μm) used in the calibration. A second limitation arises from the fact that other parameters influencing scattering (optical properties, particle shape and porosity, width of the size distribution) are not considered. The anomalous behaviour of baker's yeast can be explained in this way.

Filtration of a suspension yields two products: a cake saturated with liquid, and a filtrate with some particles. The cake consists of the cake-forming particles and a cake liquid. Cake liquid eventually becomes filtrate during the filtration process. Therefore, the clarity of the filtrate is determined by the particle content of the cake-liquid in the cake.

The particles in the filtrate originate from two sources:

- From particles in the suspension that slipped through the pores in the cake from the suspension/cake interface to the cake/filter medium interface.
- From cake-forming particles that break through after reorganisations in the cake structure, induced by gradual or abrupt changes in the operating conditions (pressure shocks).

In both cases the cake structure will play an important role. By studying the particle contamination of the filtrate, one obtains information on the cake structure. A simple approach is to describe the cake structure in terms of particle size (and porosity), and the particle content of the filtrate in terms of size and concentration.

An instrument is needed to monitor a characteristic particle size and concentration in the filtrate as a function of time. Turbidity was considered, because it is a cheap and commonly applied method to monitor filtrate clarity. The measurement of two variables requires two signals. A turbidimeter capable of measuring the forwards and sideways scattered light is necessary. This paper investigates the possibilities of using such a turbidimeter to measure particle size and concentration in a suspension.

Theoretical considerations

Characteristic size, concentration and permeability

A particle has many sizes. In filtration terms the concentration is normally expressed on a volume basis. Therefore the size to be used is preferably based on particle volume, *i.e.* the diameter of a sphere with the same volume as the particle. The characteristic size to be selected should also be related to cake structure, *i.e.* the size used in permeability calculations. The permeability K depends on the cake structure, porosity ϵ and a characteristic size d . For a homogeneous cake the relation advocated^[1] is

$$K = \frac{1}{150} \frac{\epsilon^3}{(1 - \epsilon)^2} d_s^2 \quad (1)$$

with $d = d_s$ the spherical particle with the same external surface/volume ratio as the collection of particles:

$$\frac{1}{d_s} = \frac{\sum N_i d_{si}^2}{\sum N_i d_{vi}^3} \quad (2)$$

where d_{si} is the diameter of a sphere with the same external surface and d_{vi} is the diameter of a sphere with the same volume as the particle. N_i is the number of particles with size d_{vi} per unit of volume:

$$N_i = \frac{6}{\pi} \frac{q_i x}{d_{vi}^3} \quad (3)$$

Here q_i is the volume fraction of particles with diameter d_{vi} , and x is the volume fraction of particles in the suspension. If the shape factor ϕ_s

$$\phi_s = \frac{d_{vi}}{d_s} \quad (4)$$

has the same value for all particles, the expression for d_s simplifies to

$$\frac{1}{d_s} = \frac{1}{\phi_s^2} \sum \frac{q_i}{d_{vi}} \quad (5)$$

The volumetric size d_f :

$$\frac{1}{d_f} = \sum \frac{q_i}{d_{vi}} \quad (6)$$

can be calculated if the particle size distribution (q versus d_v) is known. Since ϕ_s is difficult to measure, d_f is used as the characteristic particle size, rather than d_s .

Single particle scattering and size

The scattering pattern produced by a single particle depends on its size d and on the wavelengths λ of light used. Depending on the d/λ ratio, the following types of scattering are distinguished:^[2]

- $d/\lambda \leq 0.05$: Rayleigh scattering.
- $d \approx \lambda$: Mie scattering.
- $\lambda/d \leq 0.05$: Fraunhofer diffraction.

Although visible light contains a whole range of wavelengths, and a non-spherical particle has a range of sizes, in general Mie scattering or Fraunhofer diffraction is dominant for particles with a size of about 1 μm or larger.

Primary scattering

Mie scattering and Fraunhofer diffraction patterns are asymmetric. Most of the light is scattered in the forward direction. In that angular range ($0^\circ < \theta < 90^\circ$) the intensity of the scattered light increases with decreasing θ . This effect becomes more pronounced with increasing particle size. Thus forward scattering (*i.e.* $\theta = 12^\circ$)

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gives a higher intensity than sideways scattering ($\theta = 90^\circ$).^[3,4]

The relation between the power of the primary scattered light $P(\theta)$ and the intensity of the incident beam I_0 striking a single particle can be written in the following form:

$$P(\theta) = \alpha(\theta) S_{sca} I_0 \quad (7)$$

where $\alpha(\theta)$ denotes the scattering function, depending on the optical properties of the system, particle geometry and particle orientation. Here S_{sca} is the scattering surface of the particle, and can be interpreted in terms of a shape factor ϕ_{sca} and diameter d_{vi} :

$$S_{sca} = \pi \frac{d_{vi}^2}{\phi_{sca}^2} \quad (8)$$

For N_i particles with size d_{vi} and the same shape factor ϕ_{sca} , the primary scattering $P_i(\theta)$ becomes

$$P_i(\theta) = \alpha_i(\theta) I_0 \frac{6}{\phi_{sca}^2} \frac{q_i}{d_{vi}} x \quad (9)$$

Summing over all different sizes gives for the total primary scattering $P_1(\theta)$:

$$P_1(\theta) = \alpha'(\theta) I_0 \frac{6}{\phi_{sca}^2} \frac{x}{d_f} \quad (10)$$

According to this equation, the primary scattered light is inversely proportional to the volumetric size d_f , and proportional to the volumetric particle concentration x . However, the scattering function $\alpha'(\theta)$ also depends on the particle size d_f . Therefore Eqn. 10 can be better written as

$$\frac{P_1(\theta)}{I_0} = a_\theta(1) x \quad (11)$$

keeping in mind that the coefficient $a_\theta(1)$ depends on the scattering angle θ , particle geometry and particle size d_f .

Multiple scattering

Secondary scattering is repeating primary scattering. The basic equation for secondary scattering is clearly of the type:

$$\frac{P_2(\theta)}{I_0} = a_\theta(1) x \frac{\delta P_1(\theta)}{I_0} = \delta a_\theta(1)^2 x^2 = a_\theta(2) x^2 \quad (12)$$

where δ is the fraction of the primary beam scattered for the second time. The summation of primary and secondary scattering yields an equation of the type:

$$\frac{P_1(\theta) + P_2(\theta)}{I_0} = a_\theta(1) x + a_\theta(2) x^2 \quad (13)$$

In a similar way we can obtain the total amount of scattering by summation of all types of multiple scattering:

$$\frac{\sum P_j(\theta)}{I_0} = a_\theta(1) x + a_\theta(2) x^2 + \dots + a_\theta(j) x^j + \dots + a_\theta(m) x^m \quad (14)$$

Multiple scattering criterion

The importance of multiple scattering depends on the distance d_{opt} that the light has to travel through the suspension. Bayvel and Jones^[5] define a criterion to determine whether multiple scattering is important. When we envisage the incident light in terms of photons, the average distance a photon will travel between two collisions with particles is the 'photon mean free path' l_p ; this depends on the extinction cross-section of a particle as seen by a photon (S_{ext}) and on the number of particles per unit volume (N):

$$l_p = \frac{1}{S_{ext} N} \quad (15)$$

When the optical path length $d_{opt} \ll l_p$ then multiple scattering is unlikely, whereas if $d_{opt} \geq l_p$ then multiple scattering must occur. In practice it is suggested that multiple scattering is significant for

$$\frac{d_{opt}}{l_p} \geq 0.1 \quad (16)$$

When we express the extinction cross-section of the particle in terms of a shape factor ϕ_{ext} and diameter d_{vi} :

$$S_{ext} = \frac{\pi}{4} \frac{d_{vi}^2}{\phi_{ext}^2} \quad (17)$$

we obtain for l_p :

$$l_p = \frac{1}{\sum N_i S_{ext}} = \frac{1}{\sum \frac{q_i x}{\frac{4}{3} d_{vi}^3} \frac{\pi}{4} \frac{d_{vi}^2}{\phi_{ext}^2}} = \frac{4}{6} \frac{d_f}{x} \phi_{ext}^2 \quad (18)$$

Therefore it can be said that for a suspension with a characteristic particle size d_f , multiple scattering will become important for

$$x \geq \frac{0.15}{\phi_{ext}^2} \frac{d_f}{d_{opt}} \quad (19)$$

Outline of the method

The principle of light scattering can be applied to measure simultaneously the particle size and concentration with a turbidimeter. To obtain these two quantities, the amount of scattered light in two directions must be measured. With a turbidimeter the scattered light is measured at 12° and at 90° .

Table 1. Properties of the materials used for the calibration.

Name	Material	Colour	$d_{50}, \mu\text{m}$	$d_w, \mu\text{m}$	n	$d_f, \mu\text{m}$
Celite Standard Super-Cel	diatomite	pink	15.4	24.4	1.24	8
Celite Hyflo Super-Cel	diatomite	white	22.3	32.3	1.22	10.3
Celite 535	diatomite	white	34.3	45.6	1.19	13.8
Celite 560	diatomite	white	55	70.6	1.06	16.4
Dicalite Superaid	diatomite	pink	8.8	12.8	1.12	3.4
Dicalite Speedplus	diatomite	white	18	23.5	1.77	12.6
Dicalite 4200	diatomite	white	38.6	63.6	1.74	33.6
Dicalite 6000	diatomite	white	74.8	120.6	1.72	62.9
Dicalite 438	perlite	white	7.5	11.5	1.45	4.9
Dicalite 478	perlite	white	10	14.7	1.66	7.4
Durcal 2	CaCO ₃	white	3	4.1	0.99	0.8
Durcal 5	CaCO ₃	white	5	7.4	0.95	1.3
Durcal 10	CaCO ₃	white	8	10.7	0.91	1.6
Durcal 65	CaCO ₃	white	65	94.2	0.79	8.2
Durcal 130	CaCO ₃	white	130	175.3	1.71	90.9
Baker's yeast	organic	grey	5.9	6.9	4.91	5.9

Eqn. 14 shows the amount of scattering as a function of x , where the coefficients depend on the optical properties and on the volumetric particle size d_f . The method can only be applied if the scattering signals from the 12° and 90° detectors do not depend in the same way on d_f and x . This is the case if at one angle the influence of multiple scattering is larger than at the other angle. By calibration of samples with known concentration and particle size, the coefficients and their dependence on d_f can be obtained. It is then possible to obtain from an unknown suspension with particles with the same optical properties the concentration and particle size, by measuring the amount of scattering at two angles and using the coefficients obtained by the calibration.

Materials and equipment

In the experimental work a Monitek 210/130 turbidimeter was used. The turbidimeter originally only consisted of a 12° detector and a signal amplifier; the 90° detector was added later. The light used is white light with $400 < \lambda < 1000$ nm, while the length of the optical path $d_{opt} = 57$ mm.

The materials with which the turbidimeter was calibrated are given in Table 1. Two different kinds of filter aid were used: diatomaceous earth and perlite. The diatomaceous earth was supplied by Celite^[6] and Dicalite,^[7] and the perlite by Dicalite.^[7] Filter aids are characterised by the irregular shape of the particles and high internal porosity. Durcal^[8] is the brandname of an industrial pigment, and is characterised by a broad particle size distribution. The Monitek was also calibrated with baker's yeast.^[9] For the particle size distributions of all these materials, the data from the suppliers were used. The median particle sizes of these distributions are given in Table 1. The particle size distributions were fitted with a Rosin-Rammler distribution:

$$q^- = 1 - e^{-\left(\frac{x}{d_r}\right)^n} \quad (20)$$

For each distribution the Rosin-Rammler particle size d_r and the width of the distribution n were calculated. Then d_f was calculated by integration of the Rosin-Rammler distribution.

Results and discussion

Calibration of the Monitek 210/130

The measured signals from the forwards and sideways scattered light detectors are shown in Figures 1 and 2 as functions of the volume fraction of solids. It is seen that the forward scattered light increases linearly with concentration, suggesting little influence of multiple scattering at this angle. On the other hand, the 90° scattering is nonlinear, showing a small contribution of secondary scattering. The critical concentration for multiple scattering calculated with Eqn. 19, assuming spherical particles, gives for Durcal 2 a value of 2.1 ppm, and for Durcal 65 a value of 21.6 ppm. Both values are in the calibrated concentration range, showing that secondary scattering can be important at these concentrations. Therefore, the signals were correlated to the volume fraction of solids with the following relations:

$$S_{12} = a_{12}(1)x \quad (21)$$

$$S_{90} = a_{90}(1)x + a_{90}(2)x^2 \quad (22)$$

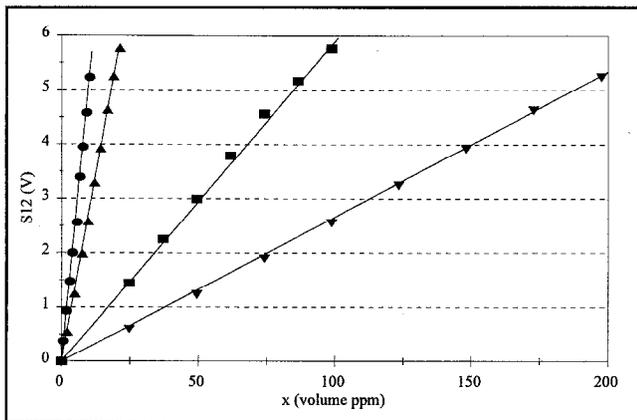


Figure 1. Signal from the forward-scattered light (12°) detector of the Monitek 210/130 plotted against concentration for Durcal 2 (●), Durcal 10 (▲), Durcal 65 (■) and Durcal 130 (▼).

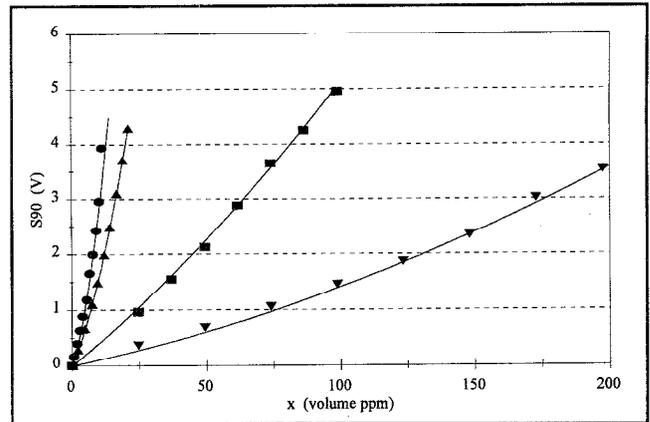


Figure 2. Signal from the sideways-scattered light (90°) detector of the Monitek 210/130 plotted against concentration for Durcal 2 (●), Durcal 10 (▲), Durcal 65 (■) and Durcal 130 (▼).

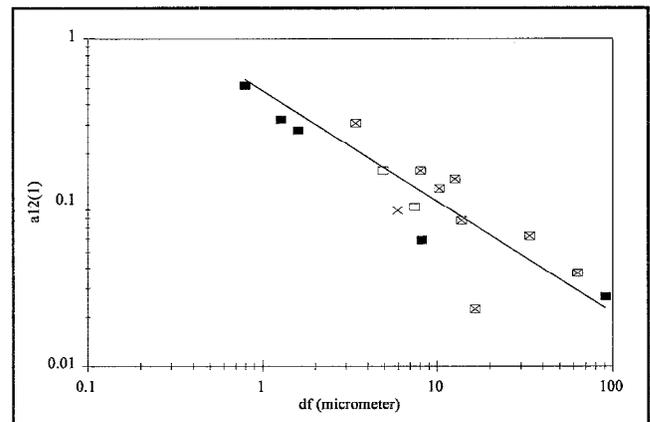


Figure 3. The coefficient $a_{12}(1)$ measured with a Monitek 210/130 against the volumetric particle size d_f .

⊗ diatomite □ perlite ■ Durcal × yeast

The dependence of $a_{12}(1)$, $a_{90}(1)$ and $a_{90}(2)$ on d_f are shown in Figures 3, 4 and 5, respectively. The following functions were found between the coefficients and d_f :

$$a_{12}(1) = \frac{0.48}{d_f^{0.68}} \quad (23)$$

$$a_{90}(1) = \frac{0.18}{d_f^{0.59}} \quad (24)$$

$$a_{90}(2) = \frac{0.02}{d_f^{1.49}} \quad (25)$$

According to Eqn. 10, $a_{12}(1)$ and $a_{90}(1)$ should be inversely proportional to d_f . Eqns. 23 and 24 show a smaller dependence on d_f . This is because of the scattering function $\alpha'(\theta)$, which is also dependent on d_f . The dependence of $a_{90}(2)$ on d_f is approximately the square of the dependence of $a_{90}(1)$ on d_f , which is in accordance with Eqn. 12. The spread in the results for diatomaceous earth is possibly as a result of the differences in the width of the distribution. The low values for yeast of $a_{90}(1)$ and $a_{90}(2)$ may be as a result of both the very narrow distribution and the different optical properties. Yeast was therefore not included in the regression of $a_{90}(1)$ and $a_{90}(2)$. Application of the method is clearly limited to substances with identical optical properties, and to applications with little variation in the width of the distribution.

Application to filtration

To test the application of the method, a filtration was carried out with Durcal 65. An AMA Cricket-filter element with a filtration area of 0.187 m^2 was used. The filter cloth is made of monofilament polypropylene with an aperture size of $200 \mu\text{m}$. The filtration

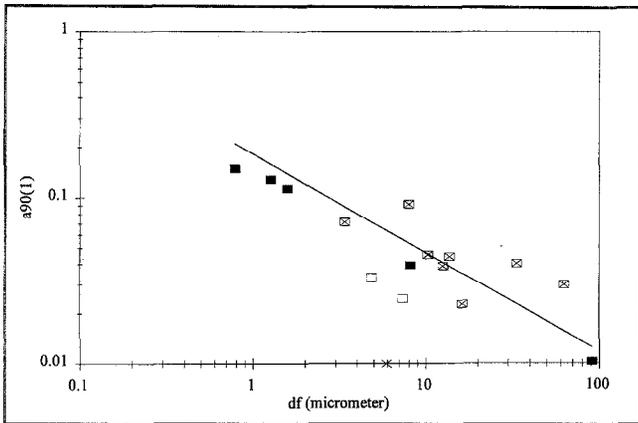


Figure 4. The coefficient $a_{90}(1)$ measured with a Monitek 210/130 against the volumetric particle size d_f .

⊗ diatomite □ perlite ■ Durcal × yeast

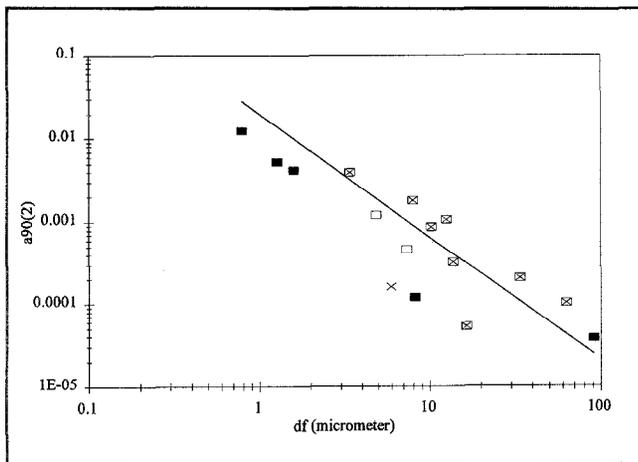


Figure 5. The coefficient $a_{90}(2)$ measured with a Monitek 210/130 against the volumetric particle size d_f .

⊗ diatomite □ perlite ■ Durcal × yeast

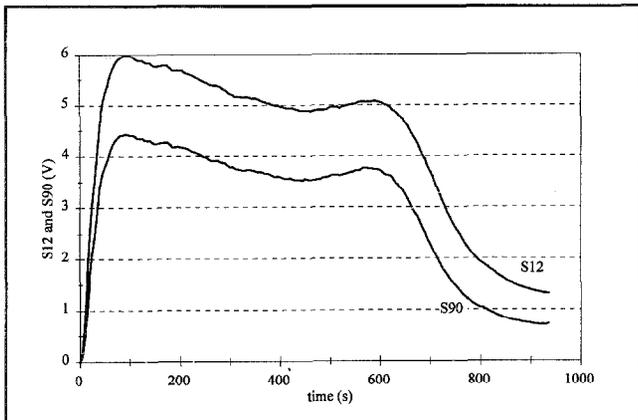


Figure 6. Signals from the 12° and 90° detectors of the Monitek 210/130 in the filtration of Durcal 65.

Filter: AMA Cricket-filter, filtration area = 0.187 m², aperture size = 200 μm. Volumetric particle size before filter d_{f0} = 8.2 μm, particle concentration before filter x_0 = 100 ppm, filtration velocity = 3.2 m/h.

velocity was 3.2 m/h. To calculate the obtained signals back to particle size and concentration, the dependence of the coefficients $a_{12}(1)$, $a_{90}(1)$ and $a_{90}(2)$ on d_f is needed. For higher accuracy only the calibrated coefficients of Durcal materials were included in the fit.

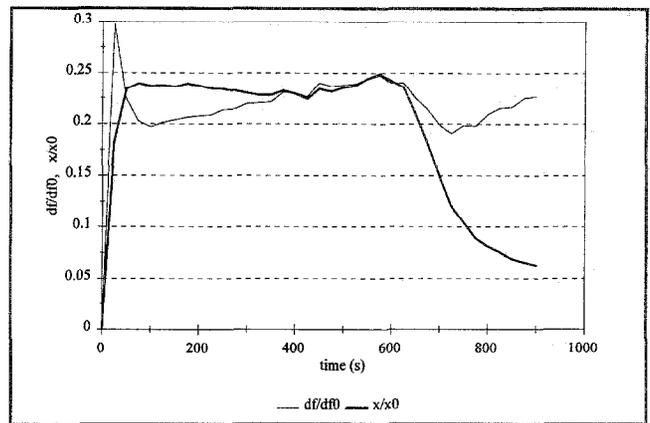


Figure 7. Particle size ratio after and before filtration (d_f/d_{f0}) and particle concentration ratio after and before filtration (x/x_0) in the filtration of Durcal 65.

Filter: AMA Cricket-filter, filtration area = 0.187 m², aperture size = 200 μm. Volumetric particle size before filter d_{f0} = 8.2 μm, particle concentration before filter x_0 = 100 ppm, filtration velocity = 3.2 m/h.

The results of the filtration experiment are given in Figures 6 and 7. In Figure 6 the two signals from the Monitek are plotted against filtration time. At the beginning of the filtration the two signals increase to a high level, showing a considerable breakthrough of Durcal particles. The signals remain at this level for some time, and start decreasing slowly after a filtration time of 600 s.

In Figure 7 the ratio of the particle sizes after and before the filter (d_f/d_{f0}) and the ratio of the particle concentrations after and before the filter (x/x_0) are plotted against the filtration time. Here d_{f0} and x_0 were 8.2 μm and 100 ppm, respectively, and d_f and x were calculated from the signals from the Monitek. After an initial breakthrough of large particles, d_f/d_{f0} fluctuates during the filtration around a value of approximately 0.22. The concentration ratio x/x_0 is constant at a value of approximately 0.24 in the first period of the filtration, and decreases slowly after 600 s. From the filtrate flow and particle concentration before the filter it can be calculated that after 600 s a particle layer has been built up on the filter, with a thickness of about 50 μm. With a d_{f0} value of 8.2 μm, this means that the breakthrough of particles with size d_f will diminish once a layer has been built up on the filter that is six times the particle size d_{f0} .

Conclusion

The particle concentration and the characteristic particle size of a suspension of particles strongly influence the scattering of light. For primary scattering the amount of scattered light will be linear with the particle concentration; however, for multiple scattering the amount of scattered light will be a higher-order function of the concentration.

The amount of light scattered also depends on the angle at which the light scattering is measured. If multiple scattering is more important for one angle than for another, it is possible to obtain both the particle concentration and a characteristic particle size by measuring the amount of light scattered at the two angles. As multiple scattering depends on the length of the optical path, the accuracy of the method can be increased by changing this length for one angle.

A drawback of the method is that the optical properties of the suspension and the width of the particle size distribution also influence the light scattering. Another drawback is that extensive calibrations are necessary.

An important application of this method lies in the analysis of filtrate clarity. Often turbidimeters are applied in the filtration industry for this purpose. Their use can be extended to this method, since they can measure the scattered light in two directions, and work in the concentration range where this method can be applied (1–200 ppm). Analysis of the filtrate in the filtration of Durcal showed that breakthrough of particles rapidly decreases when approximately six layers of particles have been built up on the filter.

Acknowledgments

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Nomenclature

$a_{\theta}(j)$	= scattering coefficient
d	= particle size (general), m
d_{50}	= median particle size, m
d_f	= volumetric diameter, m
d_{opt}	= length of optical path, m
d_r	= Rosin-Rammler diameter, m
d_s	= diameter of a sphere with the same external surface/volume ratio as the particle collection, m
d_{si}	= diameter of a sphere with the same external surface as the particle, m
d_{vi}	= diameter of a sphere with the same volume as the particle, m
K	= permeability, m^2
l_p	= photon mean free path, m
N_i	= number of particles with size d_{vi} per unit of volume, m^{-3}
n	= parameter describing width in Rosin-Rammler distribution
$P(\theta)$	= power of scattered light, W
I_0	= intensity of incident beam, W/m^2
q_i	= volume fraction of particles with size d_{vi}

S_{12}, S_{90}	= signals from the 12° and 90° detectors of the Monitek, V
S_{sca}	= scattering surface of a particle, m^2
S_{ext}	= extinction cross-section of a particle, m^2
x	= volume fraction of particles, m^3/m^2
$\alpha(\theta), \alpha'(\theta)$	= scattering functions
ϵ	= porosity
λ	= wavelength, m
$\phi_s, \phi_{sca}, \phi_{ext}$	= shape factors

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