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COMPLETE SIZE CHARACTERIZATION OF DIATOMACEOUS EARTH

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PURPOSE

Diatomaceous earth is commonly used in many manufacturing and production processes as a filtering agent. The quality of the diatomaceous earth greatly affects the effectiveness of the filter. One of the primary characteristics of determining diatomaceous earth quality is particle size. Traditional methods, like using sieves, can be tedious and may not offer enough information to completely characterize the material. Automated sizing techniques, such as sedimentation analysis or static light-scattering, are limited in scope since diatomaceous earth is not a uniform material, but a composite of shapes, sizes, densities, and colors. To completely characterize the particle size of diatomaceous earth, dynamic image analysis and dynamic light scattering (DLS) analysis are used to compliment each other to accomplish this goal. Dynamic image analysis uses shape factors to collect size data in the micron range while DLS is used to determine the size of nanoparticles that remain suspended in the medium. Testing is performed on two types of diatomaceous earth commonly used in the beer brewing industry.

MATERIALS

Samples of two different grades of diatomaceous earth (labeled DE2 and DE3) commonly used in the brewing industry are compared using dynamic image analysis (DIA) and dynamic light scattering (DLS).

DE2 **DE3**

An aliquot of each diatomaceous earth is dispersed in isopropyl alcohol, which has a higher viscosity than water and is better suited to disperse particles. An appropriate amount of the dispersion is added to the dynamic image analyzer. Using isopropyl alcohol as the suspending circulation medium, particle images are captured using a high definition camera. The images are processed and shape factors are applied to build the different types of distributions.

Another aliquot of diatomaceous earth is dispersed in deionized water and is held still for over one week, allowing larger particles to settle naturally by gravity. Any nano particles or nano fragments of the diatomaceous earth will remain suspended in the supernatant water. These nano particles or nano fragments of DE will be in Brownian motion and detectable by DLS. A small aliquot of the clear supernatant water was carefully decanted by micro-pipette from each sample and analyzed by DLS.

METHODS

Dynamic Image Analysis (DIA)

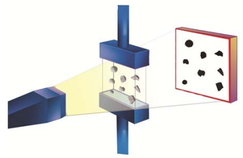
Directly measures linear and two-dimensional values and reports particle size using various definitions including equivalent spherical diameter. Direct measurement allows dynamic image analyzers to determine particle shape characteristics.

Shape Models Include:

- **Circle:** equivalent area (Heywood) diameter, equivalent perimeter diameter, bounding circle diameter, circularity, form factor, compactness
- **Ellipse:** equivalent area diameter, bounding ellipse diameter, ellipsicity
- **Rectangle:** bounding rectangle length, width, aspect ratio; rectangularity
- **Polygon**: polygon order, convexity
- **Fiber**: length, width, aspect ratio, curl
- **Irregular**: Feret length, width, aspect ratio, mean radius, smoothness
- PLUS, ability to correlate any two using a Pearson coefficient.

Analytical Assumptions and Constraints:

- Concentration is such that particles do not overlap each other during image capture.
- Limited by the magnification power of the lens
- A size distribution generated by imaging is dependent on the orientation of the particle during image capture.



Dynamic Light Scattering (DLS)

Measures back-scattering light intensity fluctuations cause by particles in Brownian motion. The intensity fluctuations are processed by the autocorrelation function (ACF) and a diffusion coefficient is calculated.

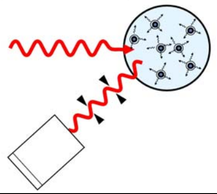
The diffusion coefficient, D, is inversely proportional to the particle size according to the Stokes-Einstein equation. The reported size is the equivalent spherical hydrodynamic diameter.

D = diffusion coefficient
k_B = Boltzmann's constant
T = absolute temperature
η_0 = viscosity of the suspending liquid
d = hydrodynamic diameter

$$D = \frac{k_B T}{3\pi\eta_0 d}$$

Analytical Assumptions and Constraints:

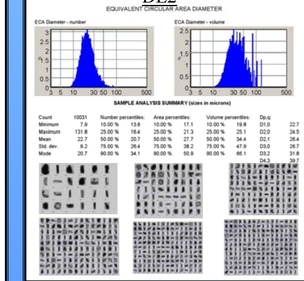
- Particles remain suspended in a liquid medium and are in Brownian motion.
- The concentration of particles is such that the suspended particles are not colliding with each other.
- Can only give size data, not concentration of particles.



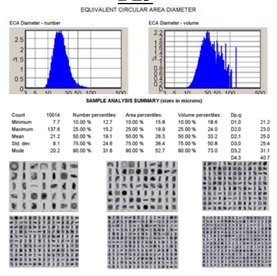
RESULTS

Dynamic Image Analysis (DIA)

DE2

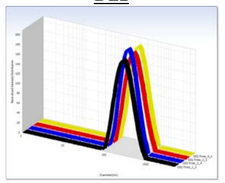


DE3



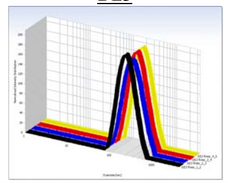
Dynamic Light Scattering (DLS)

DE2



DE2 Test	Cumulant Diameter (nm)	PI	D10	D50	D90
Diameter(nm)_1	220.8	0.231	121.4	214	389.5
Diameter(nm)_2	231	0.218	121.7	206.8	357.8
Diameter(nm)_3	221.1	0.252	119.1	209.6	380.6
Diameter(nm)_4	222.6	0.233	117.5	203.8	360.6
average :	223.9	0.234	119.9	208.6	372.1

DE3



DE3 Test	Cumulant Diameter (nm)	PI	D10	D50	D90
Diameter(nm)_1	195.2	0.241	104.7	179.9	315.8
Diameter(nm)_2	192.4	0.224	106.7	191.4	355.7
Diameter(nm)_3	193.9	0.246	105.3	184.9	333.3
Diameter(nm)_4	196.1	0.229	108.7	189	337.8
average :	194.4	0.235	106.3	186.3	335.7

CONCLUSIONS

The results from dynamic image analysis (DIA) show that the size distribution of sample DE2 is slightly larger than sample DE3, when looking at Equivalent Circular Area (ECA) diameter by number. By volume, the ECA distribution results show that DE3 is slightly larger. Visually, these results are confirmed by the thumbnail photographs of the particles. The DE3 sample had a few large particles, but was mostly fine when compared to the thumbnail photographs of DE2.

The results from dynamic light scattering (DLS) shows that the nanoparticles that remain in suspension after all larger particles have settled have a hydrodynamic diameter that is slightly smaller in sample DE3 than in DE2. This result agrees with results from DIA.

Sample DE3 has a wider distribution of particle sizes than DE2 based on DIA and DLS. Filtration of a product by DE2 and DE3 will yield a different result. DE3 has smaller nanoparticles present than DE2, which will affect the turbidity, clarity or haze, found in the finished product.

These identified nanoparticles will remain suspended indefinitely unless removed by altering their zeta potential and/or secondary filtration.

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