In-vitro Method for Quantification of Soft Focus Effect of Particulates

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Introduction

Skin imperfections such as fine lines and wrinkles are only visible on the skin when they show a contrast with the background. Microspheres, spherical particles in micron range, are known to be able to reduce this contrast and hide the skin imperfections while keeping a natural skin look due to their ability to scatter light yet let enough light through. This phenomenon is known as Soft Focus Effect or Optical Blurring. Microspheres are available in many different materials (silica and polymers), sizes and porosity. In the absence of a simple and effective method for comparing the optical performances of microspheres in various systems, the answer to the formulator question "which microsphere gives the best Soft Focus Effect?" is often only based upon experience in finished products, therefore, making it difficult to find a most suitable microsphere for each formulation.

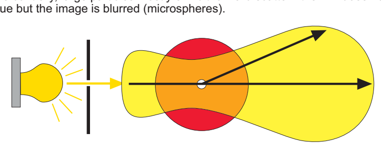
In this study, a simple in-vitro method has been developed to compare the scattering pattern of microspheres in different media, and to investigate the influence of their physico-chemical characteristics. First, a haze meter was built to measure the light scattering intensity of microspheres dispersed in a film on a glass plate. In the second part of the study, the results obtained on the haze meter were correlated with the Soft Focus Effect observed on a skin

Definitions

- Skin imperfections are visible because light is not reflected out of them : they form areas of high contrast with the rest of the skin.
- Soft Focus Effect is the capacity of particles to optically reduce the contrast and hide the imperfections of the skin surface. Light is scattered when passing through a film of cosmetic on the skin containing particles.
- Light scattering is the process by which small particles suspended in a medium of a different index of refraction diffuse a portion of the incident radiation in all directions

Mie theory and Soft Focus effect

Small particles disperse the light with approximately the same intensity at any angle. When such small particles are dispersed in a film (ie film of cosmetics on the skin), they scatter the light so that the film appears opaque (pigments).
On the contrary, large particles mostly exhibit forward scatter : the film does not appear opaque but the image is blurred (microspheres).



Small particle : mean diameter < wavelength of light (submicron)
Large particle : mean diameter > wavelength of light (Microspheres)

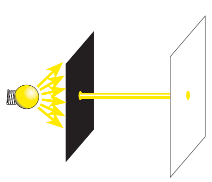
Goal

Develop a simple in-vitro method to compare the scattering ability of microspheres in different medias and to investigate the influence of their physico-chemical characteristics.

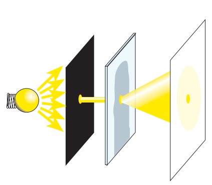
Experimental

We have developed a hazemeter, able to measure the light scattering intensity of Microspheres dispersed in a film on a glass plate. This represents a better analogy with the Light scattering which occurs on the skin than with common hazemeters where samples are measured in a liquid. In this device, a punctual light source is focused on a semi-transparent screen. Using a digital camera, picture of the image of the light source formed on the screen can be recorded and its diameter is measured using an image analysis software (ImageJ). Films of Microspheres dispersed in a media are drawn down on a glass plate using a 2 mil (50 µm) thick drawdown bar. When this glass plate is placed between the light source and the screen, Microspheres scatter the light and blur its image on the screen. The image is also analyzed and its diameter measured.
A ratio R = (Diameter of sample - Diameter of control) / Diameter of Control is then calculated and used as the scattering value.

Principle of the hazemeter



A punctual light source is projected on a semi-transparent screen. A digital picture of this image is taken and its diameter measured (Dctrl) using an image analysis software (Image J).



When a dispersion of Microspheres in a solvent, drawn down on a glass plate, is added between the light source and the screen, the light is scattered. The image on the screen is also recorded and analyzed (Ds).

The ratio of the two diameters is then calculated :

$$R = (Ds - Dctrl)/Dctrl$$

and used as a measure of the light scattering of the microsphere in a given environnement.

Microspheres

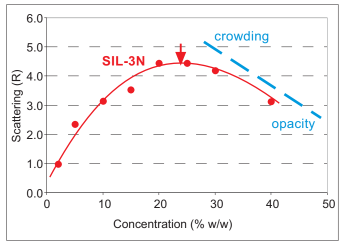
Microspheres tested in this study vary in both composition, size and porosity.
They have been tested in five different medias : Castor Oil, Mineral Oil, Isododecane, Dimethicone and as a dry film (letting the Isododecane film to dry for 2 hours).

Software

http://rsb.info.nih.gov/ij/

Influence of concentration

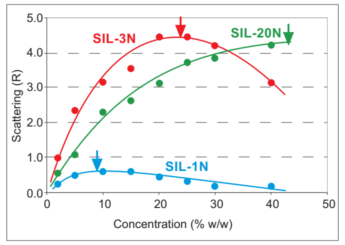
Increasing concentrations of a 3µm non-porous silica microsphere (SIL-3N) have been dispersed in dimethicone. The light scattering ratio has been measured for each concentration.



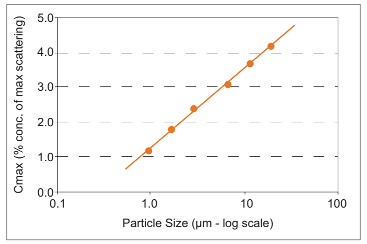
At low concentrations, scattering increases with the concentration. Above the concentration of 25% (w/w), light scattering decreases. This phenomenon is well known and described for paint as the "crowding effect" : the scattering is lower around 2 particles that are close together than around 2 particles isolated.
When the concentration increases further, the film blocks the light and becomes opaque.

Influence of particle size

Six nonporous silica microspheres, with sizes ranging from 1 to 20µm, have been dispersed in dimethicone and light scattering obtained measured at different concentrations.
Note : only 3 particle sizes are shown on the graph on the left for purpose of clarity



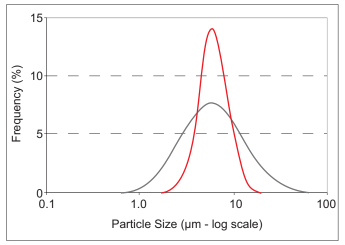
All microspheres exhibit the save pattern, scattering increasing with concentration up to a critical concentration (Cmax), where scattering start to decrease.
Small particle size (less than 2 microns) microspheres show a low maximum scattering intensity as opposed to larger particle sizes. However, Cmax is lower for small particles than for larger ones.



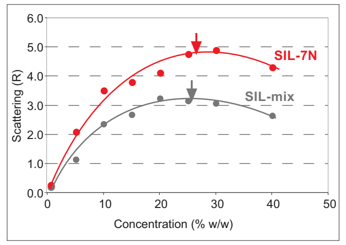
We have plotted the value of Cmax (concentration of the maximum intensity of scattering) for each microsphere as a function of particle size. This plot clearly show that higher concentration are needed for large particles to reach the highest intensity of scattering.

Influence of particle size distribution

We have compared the scattering intensity of two nonporous silica microsphere samples having the same average particle (7 µm), one with a narrow particle distribution (SIL-7N) compared to the other (SIL-mix, prepared by mixing in equal proportions 6 silica microspheres of 1, 1.75, 3, 7, 12 and 20 µm).



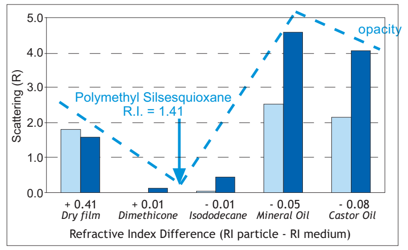
Particle size distributions of the two samples : SIL-7N (narrow) and SIL-mix (large)



The two samples tested show a similar Cmax (26-28%) : their average particle size is identical. However, the maximum intensity of scattering is lower (R = 3.2) for the sample with large particle size distribution (SIL-mix) compared to the one with narrow distribution (SIL-7N, R=4.9)
This study shows that distribution influences greatly the scattering performance of particles, but the value of Cmax seems to be controlled only by particle size.

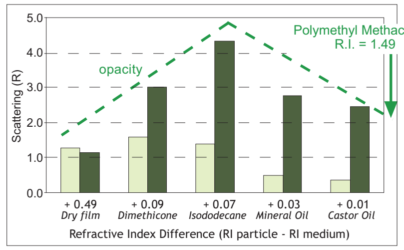
Influence of refractive index

A 4.5µm Polymethyl Silsesquioxane (silicone resin) microsphere have been dispersed at 5% and 15% in 4 solvents : dimethicone, isododecane, mineral oil and castor oil. The light scattering ratio has been measured for each concentration in each solvent. The graph bellow shows the scattering as a function of the difference between the refractive index of the particle and the refractive index of the media. A fifth test has been performed after the isododecane film had been let to dry during 2 hours.



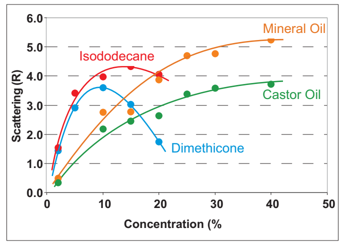
When dispersed in a solvent (Dimethicone and Isododecane) that has a similar refractive index, the polymethyl silsesquioxane particle is unable to scatter the light ("transparent"). When the refractive index difference is increased to about 0.05, the scattering is maximized. However, the maximum scattering tend to decrease when the R.I. difference is further increased, due to the higher opacity of the film.

A similar experiment has been made using a 10µm polymethyl methacylate (PMMA) microsphere, dispersed in the 4 solvents (plus dry film) at 5% and 15%.



The PMMA microsphere shows a similar result : scattering increases when the difference in refractive index increases up to about 0.07, then starts to decrease, due to the opacity of the film.

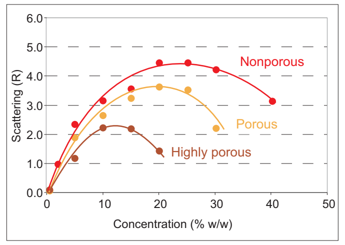
In this experiment, the 10µm PMMA microsphere has been dispersed in 4 solvents (dimethicone, isododecane, mineral oil and castor oil) at increasing concentrations. The light scattering ratio has been measured for each concentration in each solvent.



When the difference in refractive index between the particle and the media (Δ RI) is small (castor oil, Δ RI=+0.01), the maximum scattering remains low and the Cmax is very high. When the Δ RI is increased (Mineral oil, Δ RI=+0.03), the performances increase but the Cmax remains high. With a Δ RI of +0.07 (Isododecane), scattering is still high and Cmax low (best case for formulating a soft focus effect product). If the Δ RI increases further (Dimethicone, Δ RI = +0.09), the maximum scattering tends to lower, due to opacity of the film at low concentration.

Influence of porosity

In this experiment, we have compared the scattering performances of three 3µm silica microspheres, dispersed in Dimethicone at increasing concentrations. One microsphere is non porous (pore volume = 0.05 ml/g), a second one is porous (pore volume = 1.0 ml/g) and the third one highly porous (pore volume = 2.0 ml/g).



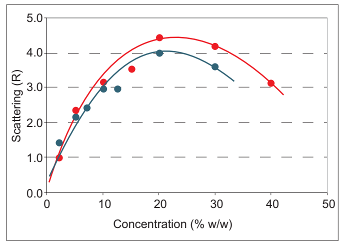
The results clearly show that porosity lowers the maximum scattering. Cmax also appears to be lower for porous microspheres : this is probably due to the concentration being expressed in weight / weight. As porous microspheres have a lower density, a given weight corresponds to a larger number of particles : therefore, crowding effect occurs at a lower concentration.

Light scattering in emulsions

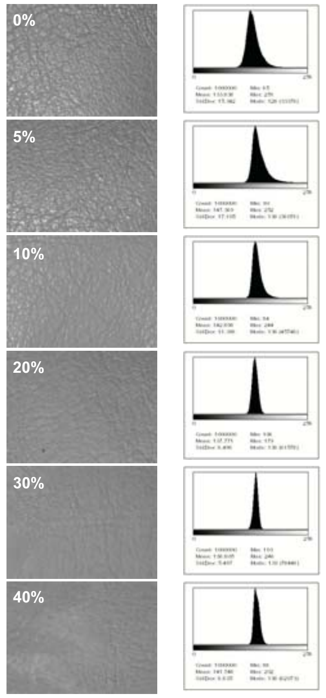
In order to correlate the results obtained with our hazemeter with actual soft focus effect observed in a formula, we have dispersed increasing concentrations (0 to 40%) of a 3µm silica microsphere in a simple emulsion :

Dimethicone	30%
Polysorbate-20	5%
Sorbitan Oleate	0.5%
PEG-150/Decyl Alcohol/SMDI Copolymer	3%
Water	qs
add SIL-3N at different concentrations	

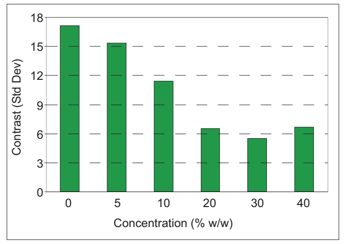
A film of each formula has been applied on to a glass plate and let dry for 3 hours before scattering was measured using the hazemeter. The following graph compares the results with those obtained for the same microspheres dispersed in Dimethicone.



These results show that there is no significant difference in the scattering ratio when this microsphere is dispersed in a solvent and when it is dispersed in an emulsion based on the same solvent.



In a second part of the experiment, the same emulsions containing increasing amounts of silica microspheres have been applied to a piece of leather. After drying for 3 hours, pictures of the leather have been taken and the pictures analyzed using an image analysis software to measure their contrast.
The graphs on the left show the distribution of grey values on each of the pictures. In this case, the larger the distribution of grey values, the more contrast the picture shows. Soft focus effect clearly reduces the contrast on each picture. The standard deviation of each contrast curves has been used as a measure of contrast and plotted (graph bellow) against the concentration.



Increasing concentrations of microspheres in the emulsion lower the contrast (ie increases the soft focus effect) up to a maximum concentration of about 20 to 30%, the 40% measurement of contrast been higher.
This pattern follows precisely the curves that have been plotted for the same concentrations of this microsphere dispersed either in Dimethicone or in a Dimethicone-based emulsion.
It clearly shows that a correlation can be established between the values obtained using the hazemeter and the Soft Focus Effect expected in formulas.

Conclusion

In the course of this study, we have developed a simple in-vitro method to assess the light scattering pattern of microspheres in order to be able to screen them for their Soft Focus Effect. This study shows that there is an optimal concentration for each microsphere in each system, with higher concentrations giving lower performance due to the crowding effect. The influence of particle size and concentration should, therefore, be studied together. The difference in refractive indices between the microsphere and the media, the particle size, the particle size distribution and the porosity of a microsphere all affect the Soft Focus Effect, and should all be considered. With this simple system, we hope to provide the formulators an effective and time saving tool for selecting a suitable microsphere to achieve the optimal Soft Focus Effect for their formulations.