Optical Observations of SOMS

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I investigated the optical properties of Swellable Organically Modified Silica when observed with cross-polarized light and a first order red plate. The samples produce a lot of color when swelled, mostly different shades of blue and orange. SOMS that was made in a puck shape displays a large array of colors before ever being swelled. This supports the idea that colors are caused by the stress the sample is experiencing causing it to rearrange its molecules which alters how light permeates the sample. The pucks are under a large amount of stress and are very unstable, so their display of color is fitting with this theory. The value of the birefringence of the substance cannot be found, as the sample is too brittle.

I. INTRODUCTION

SOMS, or swellable organically modified silica, was discovered by Dr. Paul Edmiston and Colleen Burkett of the College of Wooster in 2005. SOMS is created through a sol-gel process, but it displays properties not seen in any other sol-gel. Silica based sol-gels tend to possess the ability to absorb organic material and swell by about 15% of their original size; but SOMS displays the most extreme form of this attribute. SOMS is able to swell to around 400% of its original size. It is also unique that the sample is hydrophobic enough that it floats on water rather than absorbing it. The sample of SOMS can also be reused multiple times, by simply evaporating the absorbed organic solvent. The absorbing capability of the sample is not diminished with each use as long as all of the solvent is removed before reuse. Due to the unique structure that allows this swelling to occur, SOMS displays special optical properties.

I investigated the optical properties of SOMS using an Optiphot2-Pol polarizing microscope with crossed polarizers and a red plate compensator. When placed under the microscope, the SOMS samples displayed little to no colors under the polarizing microscope, depending on their size and how they were produced. Before swelling and at the maximum saturation point, normal samples of SOMS displayed no colors from the compensator and were only magenta, the color of the compensator when no sample is present. While swelling the sample displayed many different colors mostly in the blue and orange hues. The focus of this report is to investigate why different colors are produced under the polarizing microscope. For samples that were swelled under the microscope that possessed a flat surface a Maltese cross was observed by Lily Christman. The Maltese cross can be seen in Fig. 1. This same effect was not observed in any granular samples and only observed in small pieces of pucks that had shattered from swelling.

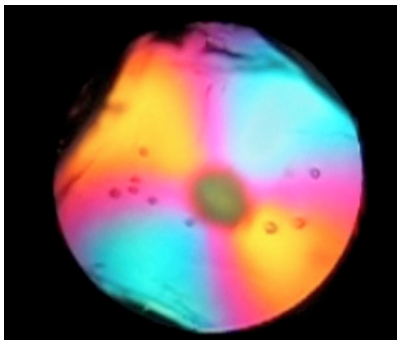

FIG. 1: As the sample swelled it displayed color in a cross pattern, known as the Maltese Cross, which vanished once the maximum swelling size was reached. The cross represents light exiting the sample at different angles. *Photo taken by Lily Christman.*

II. THEORY

A polarizing microscope is a microscope fitted with polarizers, so that a sample is illuminated by polarized light. For the microscope that I used, cross-polarizers with a tint plate compensator were used. The cross-polarizers are two polarizers oriented 90° from one another, so the light exiting the first polarizer is oriented perpendicular to the orientation of the second polarizer. One polarizer is directly above the condensing lens, which is the lens that takes in the light from the light source and converges it on the sample. This first polarizer polarizes all of the light that enters the sample to be linearly oriented. After the light passes through the sample it goes up into the eyepiece mechanism where it is polarized linearly again. So any light that was not reoriented by passing through a sample goes extinct, or when viewed through the eyepiece will look black. This is because the first polarizer orients all of the light in one direction, and the second polarizer, called the analyzer, then blocks all light not polarized in its specific direction; which do not match since they are perpendicular.

A polarizing microscope is commonly used in optical mineralogy for sample classification.[1] This is because certain optical properties are unique to minerals of spe-

cific structures. Minerals that display two different colors when viewed under a polarizing microscope with a compensator are said to be birefringent. Birefringence refers to a material possessing two different refractive indices so incident rays will be split by the two different indices. The refractive index with the highest value is called the slow axis, while the lowest value is called the fast axis. The reason this is relevant is because light will pass through the sample differently depending on the refractive index of the axis it is aligned with. This is accordance with Snell's Law

$$\frac{sin(\theta_1)}{sin(\theta_2)} = \frac{n_2}{n_1} = \frac{v_1}{v_2}, \tag{1}$$

where $\sin(\theta_1)$ is the angle of incidence, $\sin(\theta_2)$ is the angle at which the light travels through the substance, n_1 is the refractive index of the medium in which the sample is located, n_2 is the refractive index of the sample, v_1 the velocity of light in the medium, and v_2 is the velocity of light in the sample. From Eq. 1 it can be seen that two different indices of refraction would result in two different angles at which the light would exit the sample and would also affect the speed of the light within the sample. The birefringence can be classified by observing a thin section of the material with a uniform width of about 30 microns. This can not physically be done at this time with SOMS because the SOMS sample cannot be made to the specifications required without shattering, which renders the test inconclusive. The test is designed to test minerals so, if the test were successful, the results may not be exact since SOMS does not have a crystalline structure. The birefringence test is used to classify a sample based on crystalline structure.

A first order red plate compensator, referred to as a tint color plate, was used for the purpose of this experimentation to distinguish areas of interest. A compensator is a material placed just beneath the analyzer, at a 45° offset from the orientation of the polarizers, that alters the incident light to produce colors from what is otherwise observed as white light.[2] The compensator reorients a single wavelength of light which causes it to be blocked by the analyzer, so that the resultant light has a wavelength slightly longer or shorter than the original wavelength. In the case where no sample is between the polarizers, the wavelength that is not circularly oriented to match the analyzer gets blocked. For this report a 560 nm red plate compensator was used. This means that light in the green range is not permitted through the polarizer combination. With the green hue being blocked, and all other hues are allowed through making the observable color is magenta. When a sample is placed between the polarizers, the color that is observed depends on the orientation of the material as seen in Fig. 2. If the sample is oriented such that the slow axis is in parallel with the compensator's alignment, then the red color will be blocked by the analyzer and the color will appear to be light blue, which which is the color of all white light minus red. If the fast axis is aligned with the compensator, which means the slow axis is perpendicular to the compensator, the color will appear to be in the orange hue. This is because blue light is blocked, and white light appears orange minus the blue light.[3] Birefringent samples will have four points of extinction where the compensator and sample will be in opposite alignment and result in the standard green extinction. These extinction points occur when the sample is directly between the fast axis and slow axis and is thus negated. The main reason for this kind of testing is to determine the sign of the birefringence of a sample. Like the birefringence of SOMS, the sign of the birefringence of SOMS can not be found either. To determine the sign of the birefringence, the index ellipsoid is required. The index ellipsoid is a measure of the orientation and magnitude of the refractive indices. To find the index ellipsoid values such as the dielectric value of the SOMS are required, which are unknown.

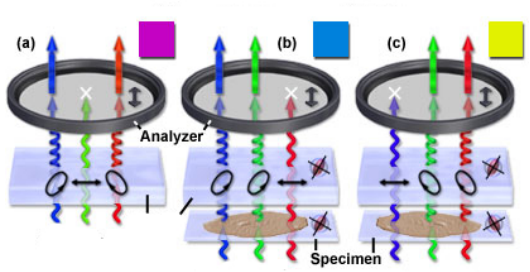

FIG. 2: The first order red plate changes the polarization of different wavelengths of white light, which causes the sample to appear colored when viewed through a polarizer. No sample results in magenta, compensator and fast axis alignment results in blue, and slow axis and compensator alignment result in yellow because of the component of white light that is blocked. *Photo taken from Olympus Microscopy Resource Center.[3]*

If the index ellipsoid were known the sample could be aligned along it and the sign of the birefringence could be found. The sign of birefringence tells whether a sample is additive or subtractive with light. If a sample is additive it means that when aligned along the index ellipsoid, the slow axis will be superimposed on the compensator, which means the birefringence will correspond to higher order birefringent colors. If the sample is subtractive it means that the fast axis is aligned and will result in lower order colors. These colors are what are used to determine the birefringent value.

There are many possible reasons as to why the SOMS produces colors when viewed through cross-polarizers with a tint plate. The most probable causes of the colors are thin film interference, stress induced optical shifts and molecular reorientation. It is believed that all three play some role in what colors are produced.

Thin film interference is an optical effect of thin materials. The most common example of thin films is a soap bubble. As the bubble floats through the air, based on

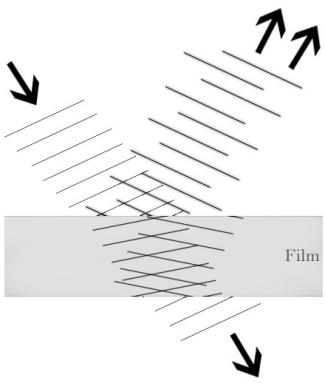

FIG. 3: The light is incident to the left. Some light is transmitted into the sample while some of the light is reflected off. The same thing occurs when the light attempts to leave the film, with some light being reflected again off the other wall of the film. The two reflected wavefronts then combine constructively or destructively.

the orientation of the light, different colors can be observed in the surface of the bubble. The SOMS sample acts in a similar way, as the term "thin film" is all relative to the incident light wavelength. Although thin film effects are usually observed in materials that are the thickness of paper or even thinner, they can be observed in materials up to a few centimeters thick.[5]

When light collides with a transparent or translucent surface, some light is transmitted through the material; but some of the light is reflected. The light is not just reflected on the surface of the material, some of the transmitted light is also reflected. Upon reaching the opposite side of the film, some of the light permeates the opposite side of the film and enters the adjacent material (for a bubble this would just be air), and some of the light gets reflected by the surface back through the material. The two sources of reflected light interfere with each other causing either a constructive or destructive interference pattern. This interference can be seen in Fig. 3. Thin film interference is possibly relevant for two reasons. the first reason is that the multi-plane reflection causes the light to exit the sample at different orientations, and orientation is very important with polarized light. A light ray that may have otherwise been negated by the analyzer, could be reflected at such an angle that it would pass through. The thin film interference could also affect the intensity of the components of the incident white light. As white light enters a sample some of the wavelengths that make it up could experience interference that could either constructively or destructively alter the intensity of each color. The sample still contains all of the colors and still appears like white light through just the polarizers but when compensate will appear a different shade.

Stress induced optical changes and molecular orientation are closely related. Stress induced optical changes is when a sample is stressed or strain and the appearance of the material changes. The reason for this change in visual properties is because on a molecular level the molecules are reorienting themselves to relieve the stress by occupying the simplest form possible. When SOMS swells it must reorient to deal with the stress of containing a larger volume between the same amount of material. The SOMS must also reorient to create room for the solvent to occupy.

III. EXPERIMENTATION

The optical properties of various sized samples of SOMS were investigated with a polarizing microscope with a first order red plate compensator with all observations done at 50X magnification. The sample sizes used ranged from fine powders up to pucks with heights averaging between 2.5 mm and 4.3 mm. Samples were placed under the microscope and observed, with the stage then rotated a complete 360° to observe all colors and extinction points.

Puck samples were placed in a Petri dish and swelled with acetone in open air until they reach their maximum swelling potential. All pucks used were new and had not been swelled before, this was proven to be insignificant in the swelling potential, but is relevant to size. The whole or only slightly damaged/cracked pucks react violently to the force of swelling. The pucks hop around the Petri dish and pieces break off the edges, with the force being great enough to cause the sample to jump high enough to invert itself.

Both shards and fine powders of SOMS were observed in addition to the pucks. These were observed by placing the samples in small quantities between microscope slides with thin brass sheets used as separators to achieve a uniform height. The slides were then held together with binder clips. The slides were then placed in a Petri dish to allow a stable base so that the sample could be rotated easier. A small space was left between the brass strips that allowed acetone to flow between the slides. All granular samples had previously been swelled.

The experiment was repeated with different solvents other than acetone to see if there was an effect on the colors and orientations observed through the microscope. This was done by using the solvents: cyclohexane, methyl alcohol, hexanol, hexane, and ethyl acetate. This was investigated by both swelling different samples with the different solvents, and by swelling the same sample with different solvents repeatedly, by heating the sample each time to evaporate off any excess.

IV. RESULTS & ANALYSIS

The Maltese cross pattern observed during swelling of normal samples of SOMS by Lily Christman was not repeatable with the same clarity in the pucks or granular

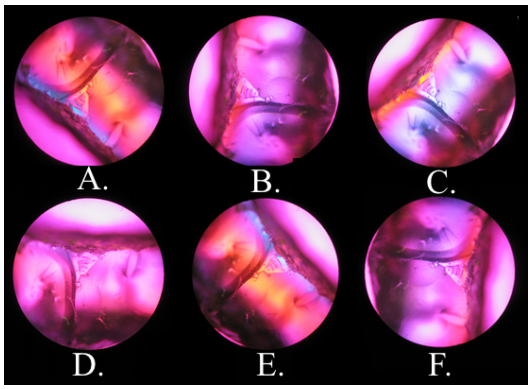

FIG. 4: Photos of a sample viewed through a crack, lengthwise, where the letters are in order or the rotation.The extinction points can be seen when the sample is oriented vertically or horizontally. When at an angle the sample displays blue and orange colors that switch every 90°

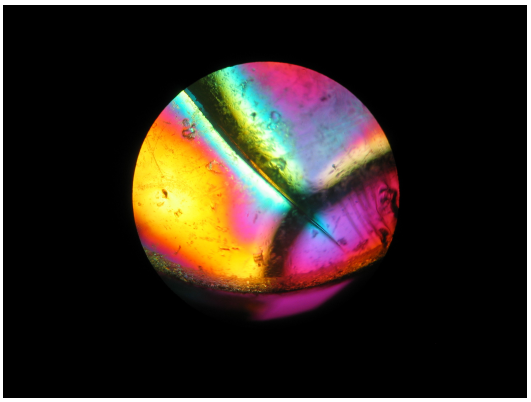

FIG. 5: The surface of a SOMS puck that had not been swelled before. The line down the center is a crack through the sample. Along the crack, the sample displays different colors (in this case, blue) than the rest of the sample. This suggests thin film interference or stress related optical shifting is a cause for the colors observed.

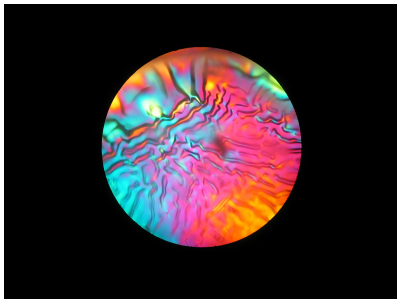

FIG. 6: The surface of a SOMS puck that had not been swelled before. The surface display ripples from the creation process. The blurred top edge is because the pucks have a different thickness around the edges due to the mold used.

samples. The Maltese cross is because light is coming into the sample at all angles, but the cross-polarizers block all but the light in their direction. So the pattern of the cross corresponds to the alignment of the polarizers. This is why the cross also can only be observed on a flat surface, as the light has to be uniform. Most commonly observed in the granules were shards exhibiting a single color, most commonly blue, and orange and sometimes yellow (which usually was surrounded by orange). The colors were very distinct and vibrant. When the microscope stage was rotated 360° the color went extinct four times as expected. The extinction points, and blue and orange transitions can be seen in Fig. 4 When a granule exhibiting either blue or orange was rotated 90° the color would switch to the opposite, so blue would become orange and orange would become blue. The biggest difference between the granules and the samples observed by Christman, is that the granules displayed colors at rest, while Christman only observed colors during swelling and shrinking. I also did not observe color in all granules, but color was observed in granules of all sizes and shapes. The idea that is was stress or the reorientation of molecules still holds true. In Fig. 5 the edge along the crack has a different color than the rest of the sample. This is most likely due to thin film in that section as it is now thinner, and stress related changes since a stress had to be applied along the fault for the fracture to occur.

The pucks displayed very interesting colors, even when not swelled. As seen in Fig. 6, the colors are very diverse and the blues and oranges can be seen at the same time. The ripples are from the surface rippling during the creation process. The edge is blurred because the outer lip of the puck has a higher height than the center.The only other time so many colors were produced at the same time was with Maltese cross. The reason the colors are present in sample that has not been swelled is that the colors are believed to correlate to the stress the sample undergoes. The pucks are created using a mold that strains the material. This strain reorients the molecules that make up the sample and because of this reorientation cause colors to be observed through the compensator. The recesses have different colors because they reorient the light at a different angle from a flat surface which causes more colors to be observable without swelling.

The use of different solvents showed no difference in the polarized colors. The order of solvents used had no effect either. The only effect noticed was that the mass of the SOMS could go below that of the original mass. This is believed to be related to solvent that is trapped in the sample being released. Different orders of solvents had no noticeable effect either.

V. FUTURE WORK

The value of the birefringence is required to do any property classifications. An attempt could be made at determining the value of the birefringence through thin

section methods. This is a very in depth method that requires a lot of time and precision, which is why it would most likely be unsuccessful. Not only can SOMS not be easily ground to a uniform thickness that thin without cracking, the samples are not crystalline so there is no consistent axis to use as a reference point to take measurements. The sign of the birefringence could possibly be discovered for the material though.

An investigation into whether temperature affected the colors produced was not attempted. There are studies to support that altering the temperature of acrylic can induce pleochroism, the property of a material displaying different colors under polarized light without the need for a compensator.[4] Further work into the optical properties of SOMS in polarized light would mostly be frivolous. Investigating the temperature dependence that may or may not have an affect on the orientation of the molecules is the only possible research that may lead to useful advances.

VI. CONCLUSION

No conclusive results were made in this experiment. The unique properties that make SOMS interesting and something worthy of study are also what make it difficult to study as there is no material for comparison. I did discover that SOMS is birefringent, but was unable to test it as the test is designed for crystalline minerals not amorphous sol-gels. The colors produced by the compensator were also identified as corresponding to the fast and slow axis, but due to the circular shape they were not useful to determine if the birefringence is positive or negative. While SOMS displays interesting optical properties they are not useful, as a field in this area does not exist yet. These properties are common and could be used to easily classify the material if it were a crystal, but since SOMS is neither a crystal nor a mineral it is not helpful.

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