Undergraduate Experiments on Qualitative Analysis of Color and Surface Modifications and Quantitative Analysis Using the Tauc Plot Method Through Ultraviolet-Visible Relative Specular Reflectance

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ABSTRACT

The study of the spectroscopy of solid materials is not as extensive compared to that of homogeneous liquid solutions. Optical properties, such as reflectance, transmittance, and absorbance, may be used for the characterization of new materials. Qualitative characterization of colored materials and quantitative determination of surface modification and band gap analysis through the Tauc plot method were obtained using visible-reflectance spectroscopy. Differentiation of reflectance peaks was observed in the spectra of colored papers and acrylic spray paints. Expected trends of higher reflectance according to the ability of a color to reflect light were also observed. Surface analysis through the reflectance method indicated that spectra of pristine and surface-modified samples showed a significant difference in signals, which is attributed to the roughening and contamination of the surface of the materials. Using the Tauc plot method on the absorbance spectrum data of an electrodeposited zinc oxide film, the calculated BGE was 3.80 eV, with a 15.15% deviation from its literature value of 3.30 eV. In addition, zinc sulfide, which was chemically deposited on glass slides, had an average band gap of 3.0 eV, with an 8.82% error against its literature value of 3.4 eV. These experiments on specular reflectance of solid materials, particularly optical and new materials, may be incorporated in undergraduate laboratory courses.

Keywords: Specular relative reflectance, surface analysis, color characterization, bandgap, Tauc Plot, solid analysis

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INTRODUCTION

Reflected light is highly dependent on the nature of the surface of a sample. There are two kinds of reflection resulting from the difference in texture and composition of the surface. Specular reflectance is observed when light is reflected from a smooth surface and at a definite angle while diffuse reflectance results from uneven and rough surfaces that tend to reflect light in all directions. (Figure 1) (Polato and Masetti 1988)

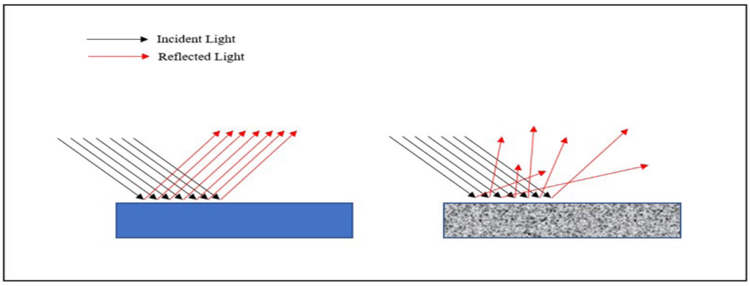

Figure 1. Specular reflectance happens when light is reflected from a smooth surface (left), whereas diffuse reflectance results from the uneven and rough surface of a sample, which reflects light in all directions (right). (Figure illustrated by Mona B. Antivola.)

Reflectance, which may be either relative or absolute, has been traditionally measured using reflectometers or reflectance attachments to spectrophotometers (Polato and Masetti 1988). Absolute reflectance calculates the true reflectance of material relative to a light source and assumes that air has 100% reflectance. Relative reflectance involves the measurement of reflectance of a reference plate using barium sulfate or an aluminum-coated mirror as the usual reference materials. Absolute specular accessories offer an option to vary the angle of incidence and may not require a calibrated mirror as the reference material, but they are more expensive than relative specular accessories. Relative specular accessories, on the other hand, require a calibrated reference mirror for baseline correction. However, due to the fixed angle of incidence, polarization may be minimized and still yield a reflectance measurement, albeit relative.

Relative reflectance accessories offer a small sample compartment that allows the analysis of smaller-sized samples but has limitations on the sample properties in that a flat sample is needed. Sample preparation is an easy step for reflectance measurements and this method of analysis prevents the sample from being destroyed, thus allowing it to be used in other parts of an experiment. Instruments have been improved over time to accommodate reflectance measurements that do not require the use of additional sampling techniques. Reflectance data is useful in the study of various properties of materials and the process is easy enough even for undergraduate students to carry out (Fernández-García 2017).

The effect of surface modification such as surface roughening and surface treatment using spray paint will lead to expected changes in the reflectance values. Another application of reflectance values, as well as transmittance, is to extract further information such as band gap energy (BGE) as shown in Figure 2. Also called forbidden energy gap (E_g), BGE quantifies the distance between the valence band, which is occupied by electrons in the ground state, and the conduction band, where the electrons move from the valence band due to excitation. This energy is simply the separation between the lowest conduction band and the highest valence band. It represents the minimum energy required for the excitation of electrons when in the state of conduction (Harouna et al. 2015).

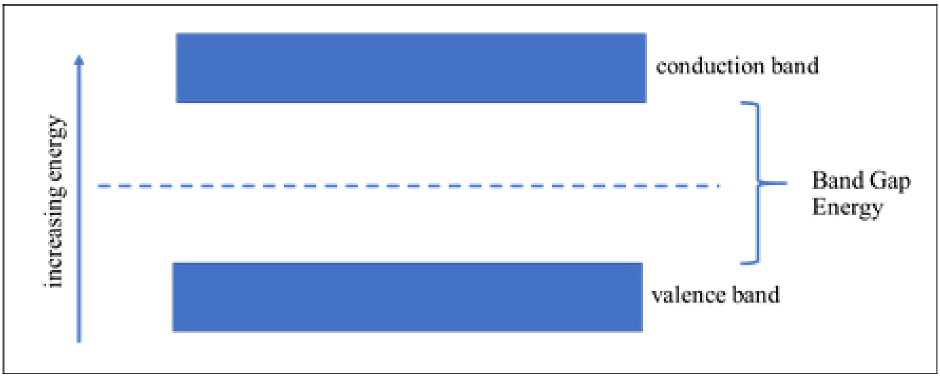

Figure 2. The band gap diagram shows the position of the valence and conduction bands. Depending on the distance or E_g, a material can be conducting or non-conducting. (Figure illustrated by Mona B. Antivola.)

The BGE dictates whether a material is a metal, semiconductor, or insulator. BGE, therefore, gives the distinct properties of a material. When there is a large distance between the valence and conduction bands, a material is insulating.

The main purpose of this study is to develop analytical and qualitative experiments for undergraduate science and non-science students for the characterization of solid materials in the range of ultraviolet-visible (UV-Vis) light using a UV-Vis spectrophotometer with relative specular reflectance accessory. It is important to develop innovative laboratory experiments using spectroscopy of solids, which is increasingly valuable in material research and development (Czegan and Hoover

2012). Several undergraduate experiments using reflectance spectroscopy were previously designed (Iyere 2000; Cordon and Lagorio 2007; Bufaroosha et al. 2020) and different equipment have been used to measure reflectance (Témun et al. 2006; Isik et al. 2017; Suli 2017; Sattar 2019; Song et al. 2019). Reflectance spectroscopy has been used for the characterization of color in different paper mediums (Havlínová et al. 2002; Harouna et al. 2015; Suli et al. 2017; Tourniéa et al. 2017; Pérez-Arantegui et al. 2018; Sattar 2019), for surface analysis (Lermond and Rogers 1955; Evans and Dennison 2012), and for the determination of BGE (Lopez and Gomez 2011; Ojeda and Rojas 2013; Vishwakarma et al. 2015; Isik and Gasanly 2017) and its quantitative analysis using the Tauc plot method (Tauc 1974; Makuła et al. 2018).

METHODOLOGY

Instrumentation

The UV-visible spectrophotometer used was a Shimadzu UV-1700 double-beam spectrophotometer, which was connected to a desktop computer and operated using the UVProbe software. Initialization of the instrument was done through the UVProbe software, after which the specular reflectance accessory (SRA) with 5° incidence angle attachment was mounted onto the sample compartment. Upon mounting the accessory, baseline correction was performed and sample analysis was done.

Undergraduate Experiments for Non-Science Majors

Reflectance of Offset Printed Commercial Colored Paper

Offset printed commercial colored papers were obtained from the brochure of reference colors from Davies® Paints (Davies Paints Philippines, Inc. 2019), which allowed for similar thickness and gloss among all samples. The papers were cut into 1 inch x 1 inch sizes and analyzed in the sample compartment of the SRA and were analyzed for three trials. The obtained spectra were overlaid in the UVProbe software and differences in spectra shape and %Reflectance values were studied. The spectra were labeled according to the color corresponding to each spectrum for easier identification; for samples with three trials, the trials were separated by different shades of the color corresponding to it. Using the UVProbe software, the peak heights and inflection wavelengths of each spectrum were determined. The spectra can be overlapped to show a comparison of %Reflectance.

Reflectance of Acrylic Paints

Glass cover slips were spray-painted to completely cover their surface. The spectra of three colors of spray paints (red paint, black paint, and white paint) were analyzed. The same procedure for the analysis of the papers was done for acrylic paints. The UVProbe software was also used to determine the peak heights and inflection wavelengths of each spectrum and an overlapped graph was made to show a comparison of %R.

Reflectance of Aluminum Foil After Roughening and Surface Treatment with Paint

Surface modification was achieved through crumpling of the foil. Qualitative analysis of optical measurements was recorded for both pristine and roughened samples. The qualitative analysis included the alteration of pristine aluminum samples through surface treatment with acrylic paint. The aluminum foil was spray-painted evenly to change the material on its surface. The surface treatment using paint on an aluminum surface is widely done in the aluminum industry for anti-corrosion.

Undergraduate Experiment for Science Majors

UV-Vis data on absorbance was used for the calculation of BGE. The optical BGE of zinc oxide (ZnO) film was obtained from its absorbance spectrum using the Tauc plot method. From the absorbance spectrum, the value of 1240 was divided by the wavelength. Additionally, the value of $(\alpha h\nu)^2$—where $h\nu$ is the incident photonic energy and α is the absorbance coefficient—was calculated by multiplying the absorbance by 2.303 and by the energy computed earlier. These two values were then used to generate the Tauc plot by plotting $(\alpha h\nu)^2$ against energy. From this plot¸ a tangent line was drawn in an area where the value of α was zero. The point of intersection between the x-axis, which was the energy, and the tangent line was the value of direct optical BGE (Tauc 1974; Makuła et al. 2018). The ZnO film was synthesized and provided by Dr. Leon M. Payawan[+].

Using chemical bath deposition (CBD) on thin films of zinc sulfide (ZnS), an inorganic semiconducting sulfide was produced. The method of CBD is particularly appropriate for use by students to produce thin films of semiconducting materials in a regular laboratory session and with standard equipment. The modified experiment is based mainly on experiments developed by Ibanez et al. (1991).

The glassware were immersed in dilute nitric acid. Half of the slide was used for depositing the semiconducting film and the experiment was done in a wellventilated hood. A water bath in a 100 mL beaker on a magnetic-stirring hot plate was set up. The reaction mixture for ZnS was prepared by adding 1.0 mL of 0.1 M thioacetamide (or thiourea) solution to 1.0 mL of 0.1 M zinc sulfate ($ZnSO_4$), followed by 1.5 mL of a 6M ammonia (NH_3) solution. The reaction mixture was placed in a 10 mL beaker containing a stirring bar and was placed in the bath. The temperature was then allowed to reach approximately 80–85 °C and was maintained for 30 minutes. The solution was allowed to cool to 50 °C while being stirred continuously. A glass slide was then placed halfway into the solution while the temperature was maintained as constant as possible at 50 °C. After an hour, the heating was stopped and the bath was cooled at room temperature. The deposition was left overnight, after which the slide was cautiously removed from the beaker using forceps. It was then rinsed with deionized water and dried under an N_2 atmosphere in a desiccator since ZnS can be oxidized into $ZnSO_4$ in the presence of moist air. After the slide had dried it was placed in the accessory and the specular reflectance spectrum was obtained.

RESULTS AND DISCUSSION

Qualitative Experiments on Offset Printed Colored Papers

The range of available colors for offset papers is so wide that it is worth expanding color analysis using this paper medium. Three batches of colored papers were prepared to test the capability of the SRA in differentiating colors.

The Reflectance of Different Color Hues

For the first batch, visually different colored papers were analyzed. The colors selected were those labeled azure blue, ivy green, chocolate brown, Cathay red, and jolly orange in the Davies® Paints brochure (Davies Paints Philippines, Inc. 2019). Multiple trials were done to ensure the repeatability of the reflectance measurement. The reflectance spectra were recorded from 900–200 nm. Figure 3 illustrates the overlay of the obtained spectra of the colored paper samples and each line is an overlay of three trials.

As observed from the spectra, an easily identifiable region is present in the 750–900 nm range. In this region, it can be observed that Cathay red has the highest %Reflectance, followed by ivy green, azure blue, jolly orange, and chocolate brown. Chocolate brown and jolly orange can be differentiated from the other colors due to their distinct spectra. Cathay red, azure blue, and ivy green converge at 800–900 nm but later diverge at the 400–700 nm area.

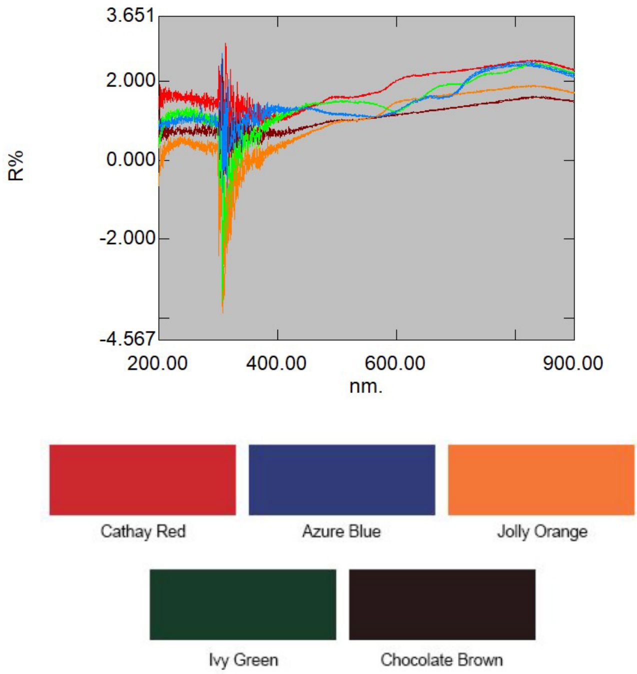

Figure 3. Reflectance spectra of differently-colored paper samples. The corresponding spectra are colored accordingly: Cathay red (red), azure blue (blue), ivy green (green), jolly orange (orange), and chocolate brown (brown). The lower figure shows reference colors for azure blue, Cathay red, ivy green, jolly orange, and chocolate brown from the brochure provided by Davies® Paints.

The characteristic peak heights of Cathay red, azure blue, ivy green, jolly orange, and chocolate brown are shown in Table 1. The reference colors were obtained from the brochure provided by Davies® Paints (Davies Paints Philippines, Inc. 2019) for easier color identification.

Table 1. Characteristic peak heights of Cathay red, azure blue, ivy green, jolly orange, and chocolate brown

Paper sample	Peak heights, nm
Cathay red	499, 630, 827
Azure blue	451, 655, 818
Ivy green	509, 681, 752, 826
Jolly orange	508, 610, 830
Chocolate brown	827

The Reflectance of Yellow and Neutral Color Shades

Two different colors and their different tones were analyzed to show the characterization that may be obtained from the analysis of reflectance spectra. The main colors were yellow and neutral. Variations of yellow are sunny day, calamansi, and yellow breeze, while neutral colors are ivory and beige. The spectra obtained from the analysis of these colored papers are shown in Figure 4.

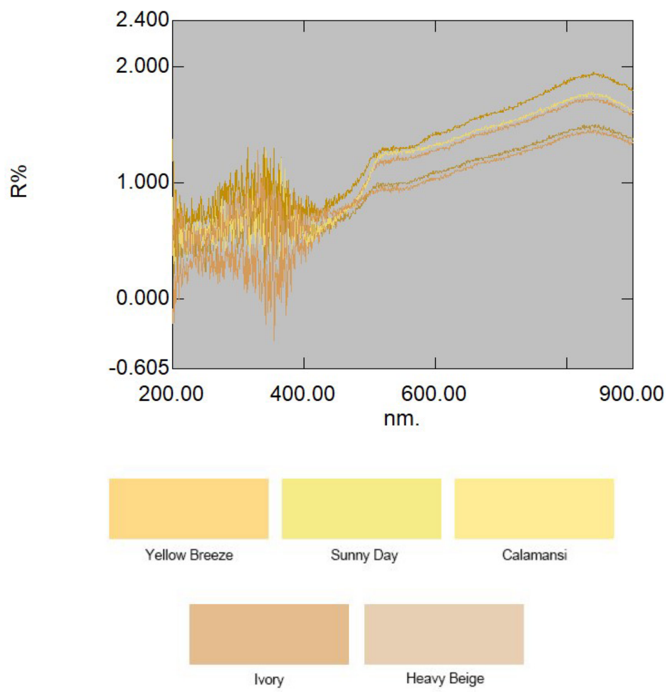

Figure 4. Reflectance spectra of different shades of yellow and neutral colors. From top to bottom, the spectra are colored according to the corresponding colors: yellow breeze, sunny day, calamansi, ivory, heavy beige. The bottom figure shows the reference colors for yellow breeze, sunny day, calamansi, ivory, and heavy beige from the brochure provided by Davies®Paints.

It is essential to identify the differences between two tones of the same color, and it can be observed that the tones of the same color can be differentiated at 522 and 835 nm with spectra having the same shape. Among the yellows, yellow breeze can be differentiated from sunny day and, while only a small difference, calamansi exhibits a lower %R compared to sunny day as well. The neutrals, ivory and heavy beige, also show a distinction, albeit a smaller difference than the former two. It should also be noted that there is a significant difference between the "parent" colors; the yellows can be grouped as all shades exhibit a higher %R reading, while the neutrals exhibit lower %R. Reference colors may vary from the real-life samples due to differences in how computers display colors. However, the differences obtained from the spectra mirror what tonal differences the human eye can detect.

The Reflectance of Shades of Pink

Similar shades of pink were analyzed to test the effectiveness of the instrument in differentiating colors. Four variations were selected for this section, namely ballerina, carnival pink, lovely blush, and pretty in pink, all of which fall under the "pink" color. The %R spectra obtained from analyzing these samples are displayed in Figure 5. Due to the difficulty posed by color-coding using similar shades of pink, the spectra are color-coded using distinctly different colors.

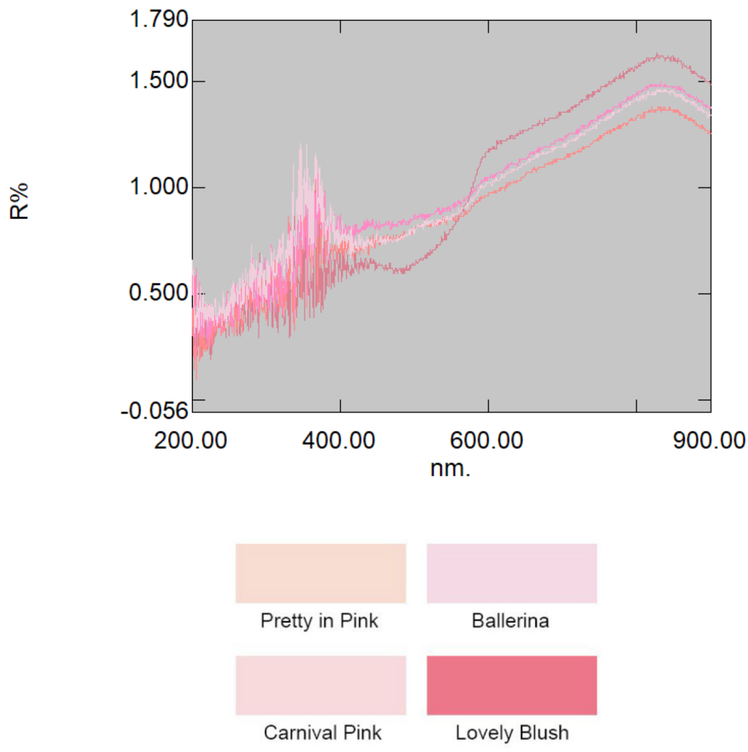

Figure 5. Reflectance spectra of different shades of pink. From top to bottom, the spectra are colored according to the corresponding colors: lovely bush, ballerina, carnival pink and pretty in pink. The bottom figure shows the reference colors for ballerina, carnival pink, lovely blush, and pretty in pink from the brochure provided by Davies® Paints.

Generally, all spectra follow the same spectrum shape as the analyses done above. Lovely blush exhibits a drastic change in %R as it goes from 900 nm to about 600 nm. It is important to note the differences in %R exhibited among the samples, as they are key to distinguishing different colors from one another.

The colors are generally close to each other and would be classified by the average person as "pink", but according to the spectra obtained, there is a significant difference that may be used to distinguish these colors from one another. Ballerina and carnival pink are colors that an average person may have difficulty differentiating due to the similar shade they exhibit, but the spectra may be able to differentiate between them. It can also be seen that throughout the three sets of colored paper samples, all samples obtained %R that did not exceed 2% reflectance. This may be indicative of how the SRA may be able to differentiate color between samples of similar composition, but may have difficulty analyzing samples that do not exhibit a significant amount of gloss.

The Reflectance of Acrylic Spray Paints

To test the ability of the SRA in differentiating red paint, black paint, and white paint, the spectra of the three colors were analyzed (Figure 6).

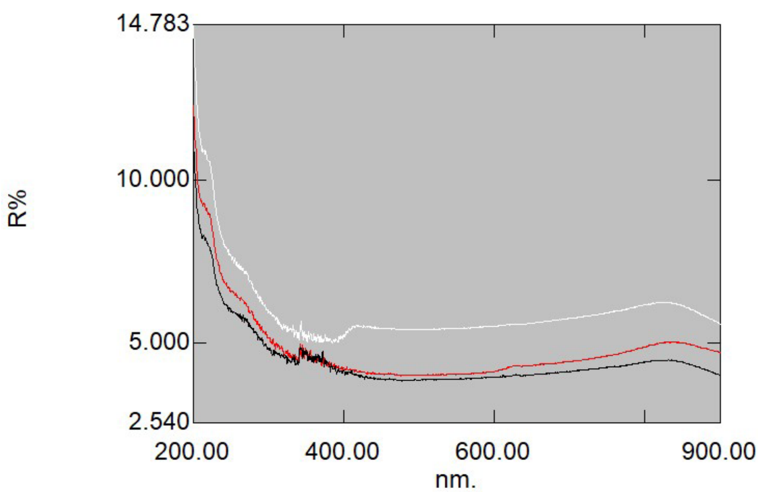

Figure 6. Reflectance spectra of acrylic spray paints. From top to bottom, the spectra are colored corresponding to the color of its sample: white, red, and black.

As observed in the spectra, the white paint exhibited the highest %R, followed by red and black. This matches the theory that white light is readily reflected, and that red and black absorb the light and thus reflect less light off its surface. In the 400–900 nm range, which is the area of concern for analysis, a significant difference between the white paint spectrum and the red paint spectrum can be observed. It should be noted that the %Reflectance obtained from these paint samples is significantly higher than those obtained from the two previous sections. This may be attributed to the gloss provided by the glass slide, as well as the gloss of the spray paint. As the length of application was approximately one second, it can be inferred that the glass slide still had a significant influence on the analysis of the samples.

Qualitative Experiments on Reflectance on Modified Surfaces

The experimental results on surface roughening by scanning the %R of a sample in its pristine form versus one with surface modification was done on the surface of aluminum. Another kind of modification was achieved by adding a layer of colored paint on aluminum foil, plastic, and mirror surfaces.

Surface Modification by Surface Roughening

Surface roughening was done on aluminum foil by crumpling the sample, thereby producing folds, dents, and possible scratches. It can be seen in Table 2 that expected changes in the measurements were brought about by changes on the surface of the material.

Table 2. Readily available samples tested for the effect of surface modification on %R

Sample Description	Initial Reading	Reading after Surface Modification
The front, shiny surface of a smooth piece of aluminum foil	19.434 %R	1.892 %R
The back of a smooth piece of aluminum foil	11.877 %R	1.611 %R

The %Reflectance scans were recorded from 900–200 nm on the spectrum mode. The spectrum of wavelength versus %R for the mentioned samples is in Figure 7 below. The difference in reflectance can be attributed to the more reflective surface of the front side of the aluminum foil.

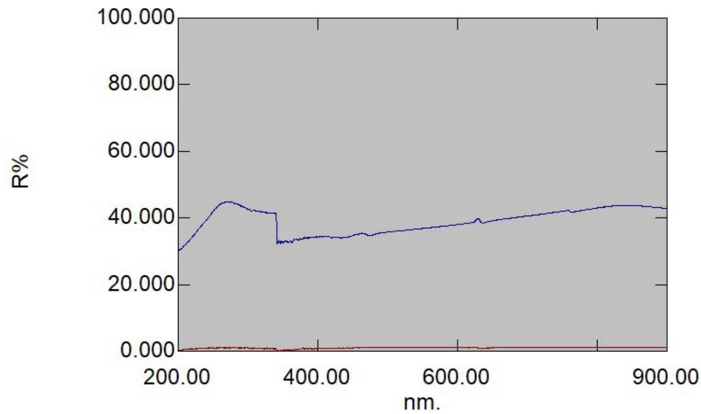

Figure 7. An overlay of the %R spectra was obtained for pristine (blue) versus crumpled (red) aluminum foil.

The Reflectance of Surfaces Treated with Acrylic Paints

Figures 8–11 show the %R spectra obtained for both the pristine form and spray-painted surfaces of aluminum foil, plastic, and a mirror. The altered surfaces of samples treated with paint showed a decrease in %R. This was expected of a homogeneous sample because its reflecting surface was covered with a non-reflecting material. Reflectance will depend on the new outer surface and its thickness. In this case, it can be said that the paint was not UV-active because of the decrease in %R**.** Furthermore, it can be noted that this method using a UV-Vis spectrophotometer with SRA can qualitatively show the effect of surface changes on the optical properties of homogeneous samples.

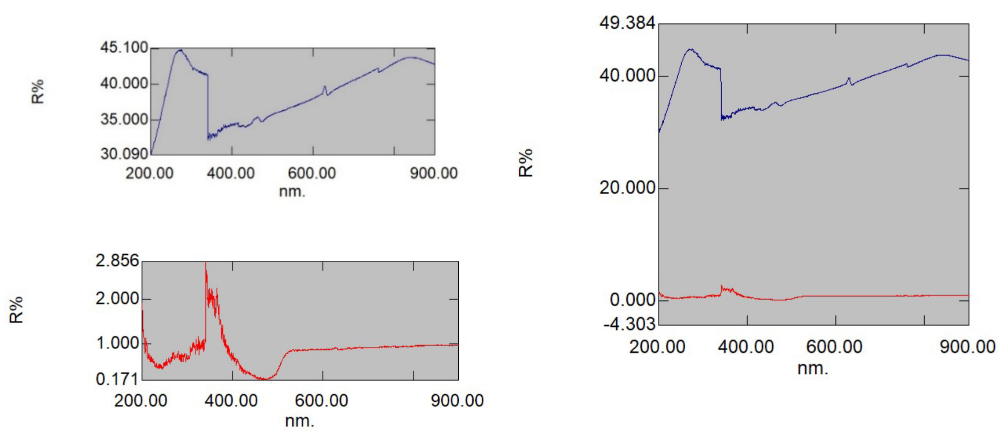

Figure 8. The %R spectra were obtained for pristine (blue) versus stained with yellow paint (red) aluminum foil. The left pictures show the individual spectrum while the right is an

overlay of the two spectra.

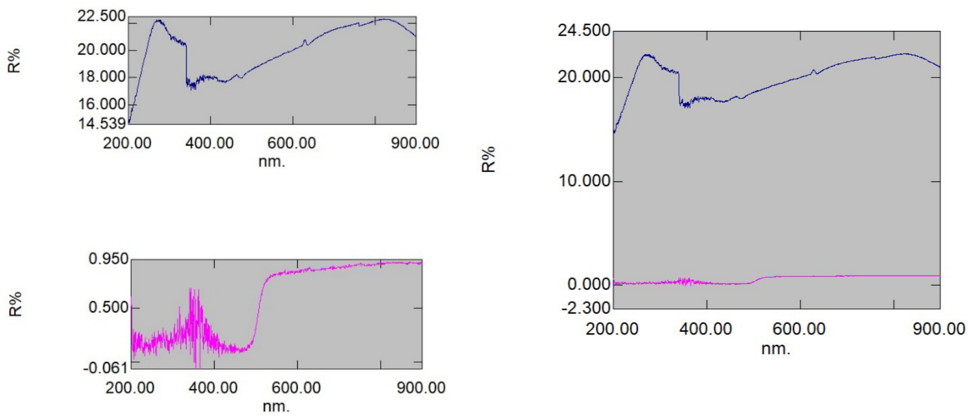

Figure 9. The %R spectra were obtained for pristine (blue) versus stained with red paint (magenta) aluminum foil. The left pictures show the individual spectrum while the right is an overlay of the two spectra.

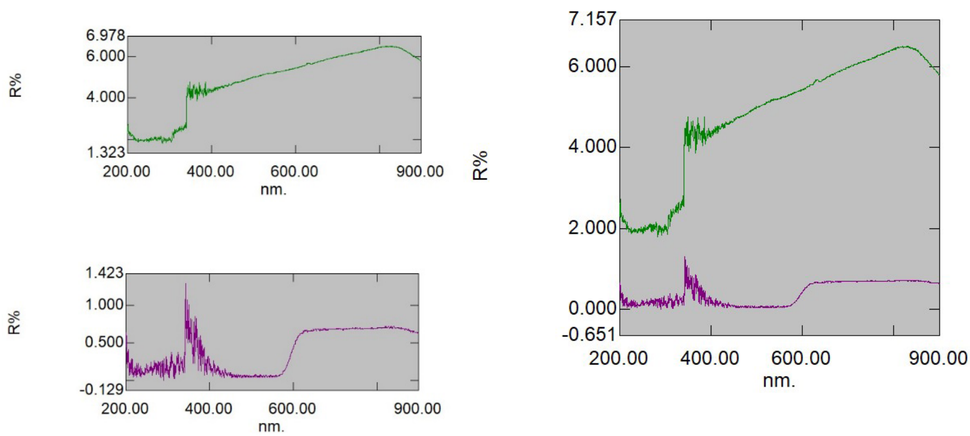

Figure 10. The %R spectra were obtained for pristine (green) versus stained with red paint (violet) thick plastic. The left pictures show the individual spectrum while the right is an overlay of the two spectra.

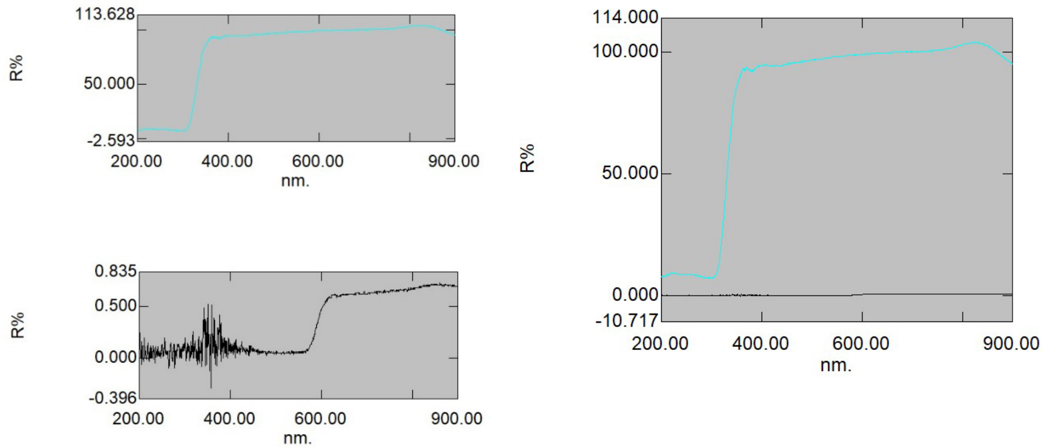

Figure 11. The %R spectra were obtained for a pristine (light blue green) versus stained with red paint (black) mirror. The left pictures show the individual spectrum while the right is an overlay of the two spectra.

Quantitative Analysis for the Calculation of Band Gap Energy Using Tauc Plot

As an application, BGE was calculated from the absorbance data generated using UV-Vis with SRA. The use of Tauc plot as the method for solving BGE limited the samples to semiconductors or nanomaterials such as ZnO doped polypyrrole nanocomposite (Olada et al. 2018**)**. Figure 12 shows one of the absorbance plots of ZnO and, from this data, the Tauc plot was generated as shown in Figure 13.

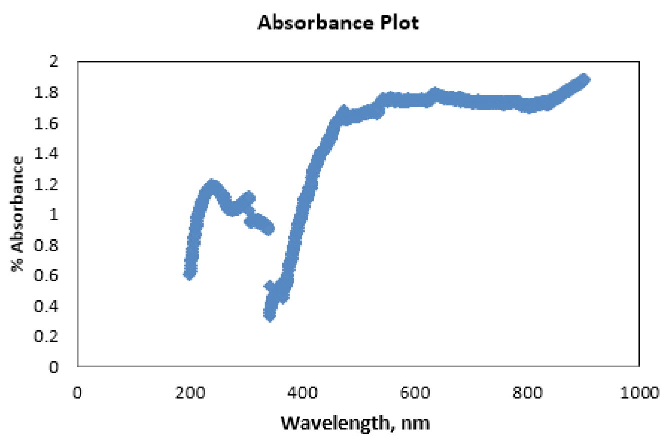

Figure 12. The absorbance plot of the ZnO sample.

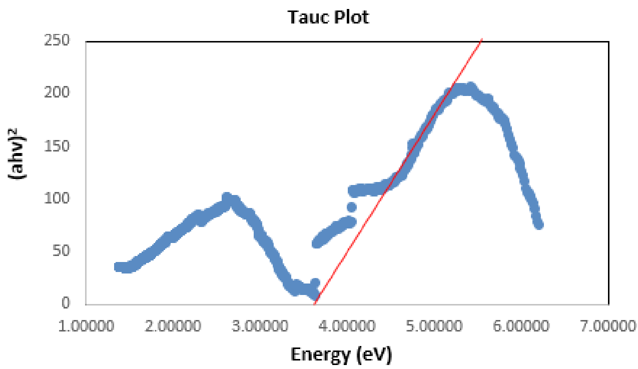

Figure 13. The Tauc plot with energy versus $(ahv)^2$ for the ZnO film sample.

The Tauc plot, which is used to calculate the BGE of films and nanomaterials, was generated by plotting $(\alpha hv)^2$ against energy. For the ZnO film samples, the average direct BGE for three trials was calculated to be 3.80 ± 0.04 eV with a 15.15 % error from its literature value of 3.37 eV (Olada and Sajedeh 2018). Knowledge of BGE has an industrial significance. As mentioned, the BGE dictates whether a material is a metal, semiconductor, or insulator. Such knowledge is used in industries, particularly those manufacturing electronic devices. The different synthetic methods of preparation may alter the band gap value of a ZnO semiconductor because structural faults may arise and alter the electronic structure (Tahir et al. 2021). ZnO is considered an insulator but in photovoltaic applications it is considered a semiconductor with BGE of 3.4 eV. Studies on ZnO have shown that it is an active material for photodegradation, and with a fast reintegration of electrons and holes, which decreases its photoefficiency.

In another activity, ZnS was chemically deposited on glass slides and its band gap was determined using an SRA (5° incidence angle) attachment. The average BGE for three trials was determined to be 3.0 eV ± 0 with an 8.82% error from its literature value of 3.4 eV (Ibanez et al. 1991). The experiment introduced ZnS as one of the most important semiconductors and it may in principle support both n- and p-doping. Also, ZnS is a low-cost, inactive compound with convenient mechanical properties such as good fracture strength and hardness (D'Amico et al. 2017). Figure 14 shows the Tauc plot of the deposited ZnS.

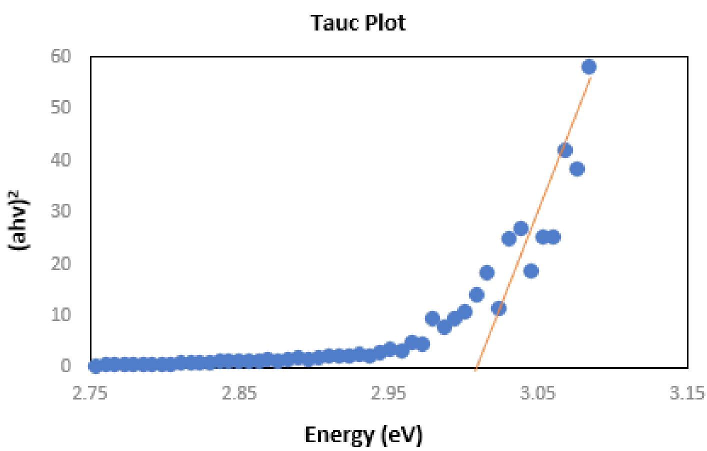

Figure 14. The Tauc plot with energy versus (ahv)2 for the ZnS film sample deposited on a glass slide.

CONCLUSIONS AND RECOMMENDATIONS

We have devised laboratory experiments for undergraduate science and non-science majors to identify and quantify the properties of solids by measuring their relative specular reflectance using a UV-Vis spectrophotometer with a relative SRA and observing a specific interaction of light with solid surfaces. Upon analysis of three different materials using the SRA, it can be concluded that color is a property that may be qualitatively analyzed. The %Reflectance range may vary depending on the parent color and material and may be key in differentiating colors for a specific material where color is the sole variable property. Generally, distinct colors have different ranges of %Reflectance, and varying color hues result in different %Reflectance readings within the given range.

It can also be concluded that an SRA is an effective tool for observing surface modifications in solid samples. Roughening causes folds, dents, and possible scratched surfaces, which showed a decrease in specular reflectance. Surface alteration by adding paint was shown to be one method to decrease reflectance and alter reflectance properties.

For the quantitative analysis, the Tauc plot method proves to be an effective tool for the calculation of BGE of thin films and nanomaterials. It is recommended to study using relative specular reflectance on materials to determine the band gap and compare with results using more expensive and advanced accessories such the integrating sphere, which is used in the American Society for Testing and Materials (ASTM) methods. Qualitative analysis of colors using reflectance spectra was shown to differentiate color hues in offset printed paper and a quantitative analysis of the colors of materials using relative specular reflectance may be carried out. Conversion of reflectance spectra to CIELAB color values and, extensively, to RGB color values has been done in previous studies using colorimeters. Specular reflectance obtained using the SRA may provide a cost-friendly alternative as colorimeters are focused mainly on color-related analyses, while SRA provides different properties to be analyzed. While this study focused on the correlation of color to specular reflectance, other studies may also be carried out to test the capability of the SRA, especially for industries that widely use reflectance data. Currently, specular reflectance is used widely in the study of thin films. Further studies may be done on nanomaterials for nanotoxicology studies and thin films for chemical sensor development. New methods of preparing the sample, such as pelletization of powdered materials, are also recommended for further studies.

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