

Mimicking Stained Glass: A Hands-On Activity for the Preparation and Characterization of Silica Films Colored with Noble Metal Ions and Nanoparticles

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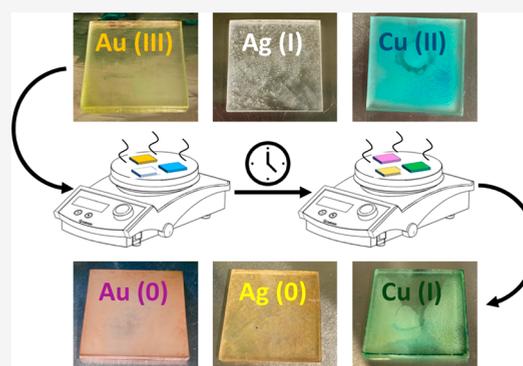
Article Recommendations



Supporting Information

ABSTRACT: We here propose a simple and fast hands-on activity requiring limited equipment, to introduce students of various levels (bachelor's or high school) and backgrounds (from science to arts) to the chemistry of color. The different colors of stained glass are replicated through the deposition, on ordinary glass slides, of silica coatings colored by addition of metal ions and nanoparticles. A silica sol is used as a matrix to embed metal ions, followed by an *in situ* reduction activated by thermal treatment on a hot plate. The formation of metal nanoparticles by this procedure induces plasmonic colors in the glass coating, thus “mimicking” the ancient procedure of stained-glass fabrication through a simple and quick method. Characterization of the colored glass by optical spectroscopy is also presented. This easily reproducible activity demonstrates basic concepts of redox reactions, chemistry of color, and plasmonic nanoparticles. Thanks to its interdisciplinary character and immediate and tangible results, this activity is well-suited for teaching laboratories for bachelor's and high school students as well as for public engagement activities.

KEYWORDS: *Crystal Field/Ligand Field Theory, UV–Vis Spectroscopy, Nanotechnology, Hands-On Learning/Manipulatives, High School/Introductory Chemistry, First-Year Undergraduate/General, Upper-Division Undergraduate*



INTRODUCTION

Colored glass has been produced since ancient times, and its production was already widespread in ancient Egypt. The techniques of glass production and processing were gradually refined, up to the colored glass panes for the production of windows and mosaics to adorn churches and other religious buildings. Transition metal ions and nanoparticles are at the basis of the creation of colored glasses. For instance, gold nanoparticles (Au NPs) confer ruby red color, and silver NPs (Ag) are responsible for yellow. Copper in its different oxidation states gives either green (Cu(I)), blue (Cu(II)), or red (Cu NPs) color. Other commonly used coloring agents include iron for green and amber colors, cobalt for blue, selenium for red, manganese for violet, and chromium for a rich green color.

The earliest techniques to prepare colored glass were based on the incorporation of metal ions within the glass matrix (*body-tinted* or *pot metal glass*), initially mainly as a result of mineral impurities such as iron and manganese. The practice of using metal nanoparticles by glassmakers, e.g., copper, silver, and gold dispersed in an optically clear matrix, is also very old and dates at least to the Bronze Age.¹ For example, incorporation of metallic silver at the glass surface from a paste containing the reagents (called “cementation”) has remained the usual method to obtain yellow stained-glass

windows from the Middle Ages through the Renaissance to the present day.² The technique of heating a glass artifact coated with a metal-based precursor was possibly invented by Coptic glassmakers in Egypt in the sixth century,¹ but it was perfected and adopted extensively only from the 14th century, giving rise to the well-known *stained glass*.

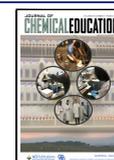
The applications of colored glass are not limited to the realm of art: optical filters, photochromic glasses, lasers, and optical fibers are only some of the cutting-edge technologies based on colored glass. Furthermore, the characterization of the optical properties of glass bears relevance to fields as varied as authenticity screening of stained-glass windows³ and glass recycling.⁴

Colored glass thus represents a highly interdisciplinary and engaging topic to demonstrate in a tangible way the chemistry of color in solid materials and the optical properties of nanomaterials. However, while the literature abounds with

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demonstrations and activities regarding the synthesis and optical characterization of noble metal nanoparticles dispersed in a liquid phase^{5–22} or embedded in a polymer matrix,^{23,24} there are no laboratory activities dedicated to colored and stained glass due to the high temperature, lengthy procedures, and safety issues involved in glass production.

We propose here a simple and fast hands-on activity requiring limited equipment, focused on the preparation of colored glass coatings by metal ions and nanoparticles. After the synthetic step, the optical properties of the prepared glass slides are characterized by UV–vis spectroscopy, but participants can also document their experience by taking photos and videos with their smartphones. This easily reproducible activity demonstrates basic concepts of redox reactions, chemistry of color, and plasmonic nanoparticles. To facilitate the integration of this activity into arts curricula, Section S1 reports historical background about the use of nanoparticles in artistic artifacts. Thanks to its interdisciplinary character and its immediate and tangible results, the activity is well-suited for teaching laboratories for bachelor's students as well as for public engagement activities.

EXPERIMENTAL OVERVIEW

This hands-on activity is based on the deposition of silica coatings colored with metal ions and metal nanoparticles. The latter are produced by *in situ* reduction, which can be monitored by a change in color of the film. Different colors are observed depending on the metal oxidation state.

Glass color induced by metal ions can be explained on the grounds of the ligand field theory, which also explains the behavior of metal complexes in solution. The metal ions exhibit different colors depending on their electron structure²⁵ and electronic configuration, which in most cases shows *d* orbitals that are only partially occupied. When metal ions form a complex, *d* orbitals change their energy state (loss of degeneracy) due to the static electric field produced by the surrounding anionic neighbors, with orbitals closer to the ligands increasing their energy with respect to those which are further away. This generates an energy splitting that depends on the nature of the ion, its coordination geometry, and oxidation state. As this energy splitting generally falls within the range of visible light, electrons in *d* orbitals can thus be excited by absorption of photons of visible light. The color of pot metal glass can be explained by *d*–*d* transitions of the metal ion coordinated by oxygen atoms within the glass matrix.³

When metal ions change their oxidation state to zero, metal nanoparticles are formed. The latter often show a different color compared with their ionic counterparts. The very strong optical absorption of metal NP-containing glass depends on localized surface plasmon resonance (LSPR). LSPR is a phenomenon characteristic of certain metal nanoparticles, such as gold or silver NPs: It consists of a resonant collective oscillation of the conduction electrons of the metal nanoparticles stimulated by an incident light. When the incident photon frequency resonates with the localized surface plasmon, a wavelength-selective absorption with extremely large extinction coefficients is observed.^{26,27} The absorption wavelength does not depend only on the metal species but also on the shape and size of particles, as well as on the composition of the surrounding medium.²⁸

In this activity, colored glass was obtained by deposition of a surface coating composed of a silica matrix with embedded

color-inducing metal ions and nanoparticles. The silica layer was deposited by spin coating of a colloidal solution (or sol) of silica prepared by a sol–gel method.^{29,30} The silica colloidal particles contained in the ethanol-based sol can form a uniform silica coating at the surface upon solvent removal, without the need for high temperature heat treatments. This colorless, transparent surface coating is then colored by the deposition of a few drops of a solution of a metal salt on top of the freshly deposited silica layer. The positively charged metal ions tend to adsorb at the negatively charged silica. The final color of the coating will thus depend on the nature of the metal ion: NaAuCl₄ solutions lead to yellow layers, and AgNO₃ forms white coatings, while Cu(CH₃COO)₂ results in blue films. The color of the coating can then be changed by altering the oxidation state of the metal centers. In particular, *in situ* reduction can be obtained simply by heating the coating on a hot plate at ca. 300–350 °C.⁵ The whole process is represented in Figure 1.

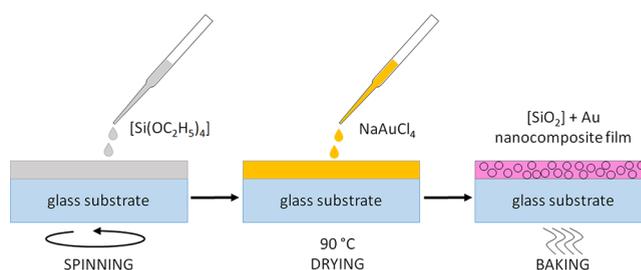
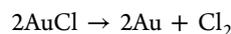


Figure 1. Schematic representation of the Au-modified silica film preparation.

The thermally induced oxidation of the organic components of the silica layer (e.g., the nonionic surfactant stabilizer) induces the reduction of the metal ions, which can be observed as a change in color of the film: blue Cu(II) species are reduced to green Cu(I), while Au(III) and Ag(I) ions are reduced to metal nanoparticles, showing pink and yellow colors, respectively. In the case of gold, Au nanoparticles could be produced also via thermal degradation of HAuCl₄³¹ according to this series of reactions that lead to metallic Au around 320 °C:



On the other hand, organic residues play a main role in the reduction of the other metal salts, as for instance AgNO₃ is known to require much higher temperatures (ca. 500 °C) to thermally degrade to metallic Ag.³¹

The presence of the silica layer plays a pivotal role in the preparation of metal nanoparticles: The not fully condensed silica matrix can adsorb metal ions, favoring their dispersion and thus preventing aggregation during particle formation. Similarly, a crucial step for metal nanoparticle formation during stained-glass preparation is the diffusion of noble metal ions within the silica matrix: during the annealing step, high temperatures favor the ionic exchange between noble metal ions with alkali metals initially contained in the glass.¹⁶

MATERIALS AND INSTRUMENTATION

Tetraethyl orthosilicate, ethanol, a 1 M hydrochloric acid aqueous solution, Pluronic P123 (poly(ethylene glycol)-*block*-poly(propylene glycol)-*block*-poly(ethylene glycol)), average molar weight 5800 g mol⁻¹, 2-propanol, acetone, concentrated sulfuric acid (95%), and metal salts (NaAuCl₄, AgNO₃, Cu(CH₃COO)₂) were purchased from Sigma-Aldrich. Water that passed through a Milli-Q apparatus was adopted for glass substrate cleaning. It should be noted that deionized and distilled water can be used as replacements for Milli-Q water. Pyrex glass slides (3 cm × 3 cm) were used as substrates; square supports of smaller size can be used provided that the spin coater is equipped with a sample holder for small fragments.

Cleaning of the supports and solution preparation was performed with an ultrasonic bath. Silica film deposition was carried out by a spin coater (SPS-Europe). The colored film deposition requires an oven and a heating plate. The optical properties of the prepared films were then characterized using a UV-vis spectrophotometer (Shimadzu UV-2600) equipped with an integrating sphere.

HAZARDS AND SAFETY PRECAUTIONS

During the activity, lab coats, safety goggles, and gloves should be worn. Silver nitrate can stain the skin. The glass substrate cleaning should be performed in advance by a lab technician able to handle concentrated H₂SO₄ solution: After the cleaning procedure, the glass slides must be rinsed and then immersed in Milli-Q water and dried just before the activity. The HCl 1 M solution is corrosive. Tetraethyl orthosilicate is a flammable eye irritant compound and may cause respiratory irritation if inhaled. Sol preparation and salt deposition via drop-casting should be performed under a fume hood due to the use of ethanol, a volatile and flammable organic solvent. The hot plate must be used under a fume hood, and care must be taken to avoid placing glass slides still wet from ethanol solutions on the hot surface; the hot glass slides should be handled with heat-resistant tweezers, and Pyrex glass is preferred to reduce the chances of slide breakage due to thermal shock.

EXPERIMENTAL PROCEDURE

Silica Sol and Surfactant Solution Preparation

The silica sol was prepared at room temperature, following a procedure previously reported.²⁹ 25.4 mL of ethanol was mixed with 2.2 mL of a 1 M HCl aqueous solution and 4.9 mL of tetraethyl orthosilicate (TEOS) and stirred for 1 h. Table 1 summarizes the reagent amounts used for sol preparation.

The prepared sol was then aged at least for 24 h at room temperature; hence, it is recommended to perform this preparatory step at least 1 day before the laboratory activity. The silica sol can be stored in a closed glass container in environmental conditions for several weeks. However, we

Table 1. Reagents for the Silica Sol Preparation

reagent	molecular weight (g mol ⁻¹)	density (g mL ⁻¹)	volume (mL)
tetraethyl orthosilicate	208.33	0.933	4.9
ethanol	46.07	0.789	25.4
1 mol L ⁻¹ HCl aqueous solution	36.45	1.02	2.2

recommend to avoid prolonged storage that could change the sol viscosity resulting in poorer film quality.

The surfactant solution should also be prepared in advance: 25 mL of a 17 wt % Pluronic P123 solution in ethanol was prepared. Pluronic P123 is a linear chain surfactant which can be easily degraded around 300 °C: If necessary, it can be replaced with other surfactants of the Pluronic family.

Preparation of Metal Salt Solutions

The salt solutions were prepared using ethanol as solvent. The reagent amounts and solution concentrations are reported in Table 2. The noble metal salt solutions can be prepared in

Table 2. Noble Metal Salt Solution in Ethanol

reagent	molecular weight (g mol ⁻¹)	concentration (mmol L ⁻¹)
NaAuCl ₄ ·2H ₂ O	397.80	17.5
AgNO ₃	169.87	54.9
Cu(CH ₃ COO) ₂ ·H ₂ O	199.65	58.4

advance and stored in the dark. It should be noted that AgNO₃ and HAuCl₄ are photosensitive compounds: For instance, exposing the AgNO₃ solution to ambient light for few hours causes appreciable photochemical reactions, observed as a change in color and by precipitate formation.

Glass Slide Pretreatment

It is recommended that the glass substrate pretreatment is performed before the laboratory activity. The substrates were 3 cm × 3 cm Pyrex glass slides. The substrates (6 glass slides for each group) were cleaned sequentially with 2-propanol, acetone, and Milli-Q water by sonication in an ultrasonic bath. After this procedure, the glass substrates were immersed in a concentrated H₂SO₄ solution (95% in weight) for 30 s, and then rinsed with water. As an alternative, other oxidizing treatments, such as plasma cleaning, can be performed to remove surface contamination and favor film adhesion. The cleaned substrates should be stored immersed in Milli-Q water for a maximum of 24 h and dried right before the laboratory activity.

Spin-Coating Deposition of Silica Sol

After 24 h of aging, the sol was mixed with the surfactant solution: 1 mL of Pluronic P123 solution was added to 9 mL of silica sol; this was stirred for few minutes in a vial at room temperature.

The mixture of Pluronic P123 and silica sol was deposited via spin-coating on the pretreated glass slides using the following parameters: time 20 s, rotation 2000 rpm, acceleration 500 rpm/s. The spin-coating step deposits a uniform film at the surface, removing the solvent. As an alternative, the silica sol/Pluronic P123 mixture can be deposited on the glass slide by drop-casting, and then, the solvent must be removed by drying the glass slides in an oven at 90 °C for around 10 min before the deposition of the noble metal salt solutions.

Drop-Casting Deposition of Metallic Salts and Nanoparticle Formation

Right after the deposition of the silica coating, the metal salt solutions were deposited via drop-casting on the silica film: Around 20–25 drops of solution were deposited over each glass substrate. The films were then dried in a stationary oven

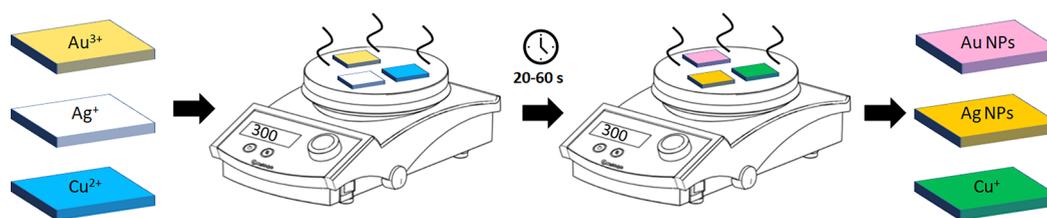


Figure 2. Schematic representation of the color change upon *in situ* reduction of metal ions by thermal treatment.

at 90 °C for about 5–10 min. After drying, the films present the color characteristic of each metal ion.

For each metal, two films were prepared: One should be characterized as such, while the other is prepared to be reduced by thermal treatment. The comparison between the two films (as-prepared and reduced) provides a more striking visual contrast.

In order to promote the *in situ* reduction of the metal ions, a film at the time was then placed directly on a hot plate (ca. 300 °C) using heat-resistant tweezers. The reduction can be monitored by a color change of the film: Au(III)-colored films turn from yellow to pink due to Au NP formation, the Ag(I)-colored ones from white to yellow upon reduction to Ag metal NPs, and the blue Cu(II)-colored films turn bottle-green upon Cu(I) formation. Figure 2 schematizes the *in situ* reduction process.

Optical Analysis

The optical properties of the colored films were analyzed by UV–vis spectrophotometry. Spectra were collected in reflectance mode, by fixing the glass slide over an aluminum reflective plate (as shown in Figure S1). The spectra were collected before and after the hot plate reduction procedure, between 300 and 800 nm. The background subtraction was performed against the plain aluminum reflective plate.

RESULTS AND DISCUSSION

Student Experience

The full experience (film preparation on six glass slides and optical analysis) takes around 1–2 h, depending on the laboratory experience of the participants. The activity can be carried out individually or in two-person groups. To speed up the activity, glass pretreatment and solution preparation were performed beforehand, as detailed in the [Experimental Procedure](#). To minimize idle time, each group deposited one film at a time: When the first one was put in the oven to dry, a second one was deposited, and so on. All of the participants carried out the activity successfully and reported their results with photos and videos.

Film Deposition

After mixing the silica sol with the P123 solution in a closed glass bottle, each group sampled 1–2 mL of a metal salt solution, gold, silver, or copper, in a closed glass vial. Then, the silica sol–P123 mixture was deposited via spin-coating on the glass substrate (8–9 drops). An image of the resulting film is reported in Figure S2. The film-coated glass plate was placed on a glass Petri dish for easy transfer, and under a fume hood, 20–25 drops of the salt solution were immediately deposited on the fresh silica film, until the whole surface was covered. Afterward, the glass plate on an open Petri dish was carefully transferred into an oven to remove the solvent. At this stage,

the student must pay attention that the deposited salt solution does not overflow from the glass surface.

Nanoparticles Formation and Optical Analyses

The dry films could be characterized as such or reduced on a preheated hot plate (placed under a fume hood). The reduction takes place in around 20–60 s (depending on the metal salts and the ion concentration), and it can be followed by the color transition of the film (Video S1): Figure 3 reports

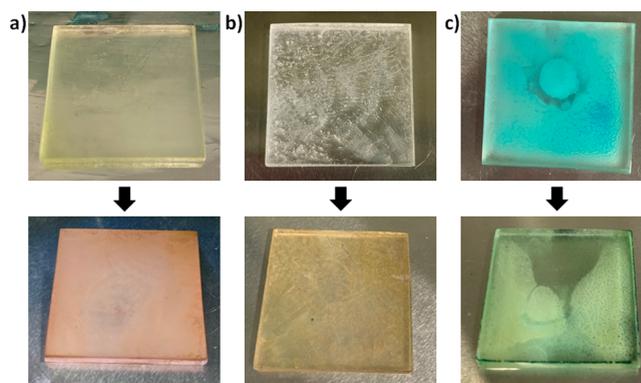


Figure 3. Photos of the silica films colored with noble metal salts before and after reduction: (a) gold, (b) silver, and (c) copper. The glass slides in the picture (3 cm × 3 cm) are placed on an aluminum plate to better appreciate their colors.

representative examples of the Au-, Ag-, and Cu-based films before and after reduction. Local excesses in the deposited salt can result in a foggy appearance of the film. While drop-casting followed by drying in the oven minimizes the necessary equipment and preparation time, the homogeneity of the resulting films is highly dependent on the manual skills of the operator. Multiple layer depositions by spin-coating can be adopted as an alternative to improve the film homogeneity. Care must be taken to remove the film from the hot plate when it starts to change color: Heating for too long can result in carbonization of the organic residues, leading to a brownish color. Before optical analyses, the hot glass slides must cool down for few minutes.

Finally, the reflectance spectra of the prepared films were collected with a UV–vis spectrophotometer by attaching the glass plate on a reflective aluminum support. Figure 4 reports representative reflectance spectra of as-prepared and reduced films, along with the reference spectrum of an uncoated glass slide. The color observed depends on a combination of the reflection (scatter) and absorption of specific wavelengths of visible light by the sample. An attenuated reflectance with respect to the bare glass substrate corresponds to light absorption in that wavelength range. If the light absorbed is in the visible region, the resulting reflected light will be characterized by a color. The observer sees the light that is

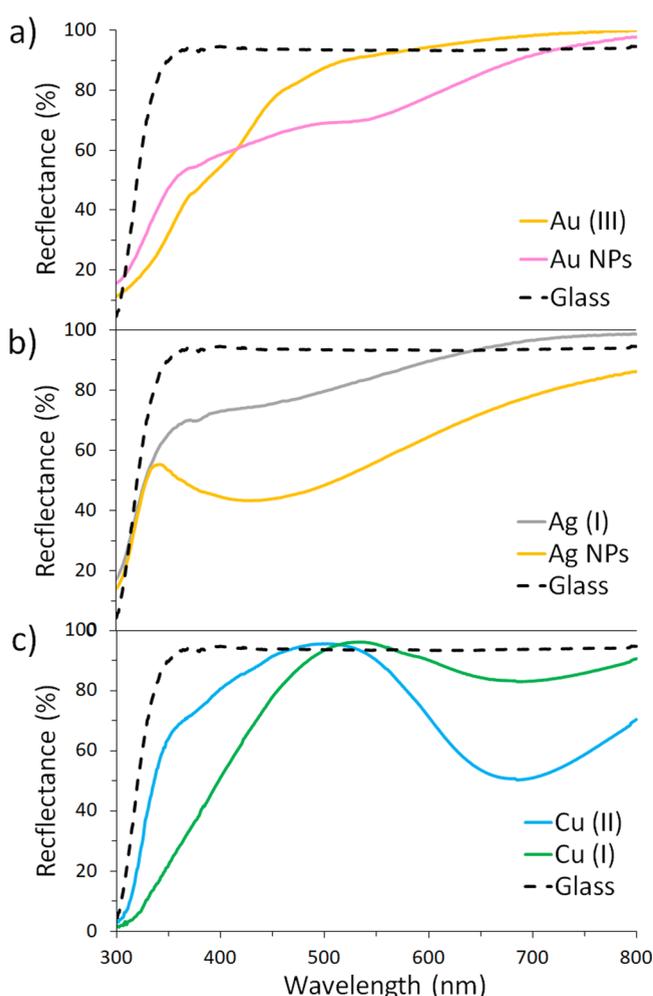


Figure 4. UV-vis reflectance spectra of (a) Au, (b) Ag, and (c) Cu films before and after reduction. The spectrum of the uncoated glass slide is also reported as reference. The spectra were recorded in reflectance mode with an aluminum plate as reference and behind the colored glasses.

reflected (or transmitted), thus perceiving the complementary color to the absorbed wavelengths (color wheel in Figure S3).^{32–34} For example, if the stained glass absorbs in the blue region, it will have an orange/yellow color. It should be noted that transmittance measurements can also be performed, using suitable sample holders,³⁵ if available spectrophotometers have no integrating sphere.

Figure 4a shows the UV-vis spectra of gold-containing films: the spectrum of the Au(III)-containing sample has an intense absorption centered around 450 nm (blue/purple region), which is responsible of the yellow/orange color of the coating, while the reduced film containing Au NPs absorbs mainly in the green region (LSPR band around 550 nm), thus resulting in a pink color. The silver-modified glass spectra are reported in Figure 4b: While Ag(I) addition leads to minor light absorption in the visible region resulting in a whitish film mainly due to scattering phenomena, the Ag NP-containing coating shows an intense absorption in the blue/purple region (LSPR band centered around 450 nm), that gives rise to the yellow/orange coloration of the film. It is noteworthy that Au(III) and Ag NPs, both with a yellow color, are characterized by different UV-vis reflectance spectra: The glass composition can thus be related to the optical properties

of the films by spectroscopic analyses.³ For what concerns copper stained-glass plates (Figure 4c), the Cu(II) spectrum shows a main absorption in the red region (around 650–700 nm), which gives rise to a blue-green color that can be attributed to d–d transitions of Cu²⁺ in distorted octahedral field surrounded by oxygens.³⁶ On the other hand, Cu(I) species mainly absorb in the ultraviolet region,³⁷ while the bands at 320–370 and 400–440 nm can be attributed to charge transfer bands of O–Cu–O and Cu–O–Cu complexes,³⁶ thus resulting in a greenish color. No bands in the 565–570 nm region can be appreciated; this is the characteristic region of the plasmon resonance of Cu nanoparticles.³⁸ It is widely recognized that copper exists as Cu(II) in equilibrium with Cu(I) in a glass melt and that this equilibrium is ruled by the oxygen fugacity.³⁸ Only if severe reducing conditions are achieved (either by controlling the kiln atmosphere or thanks to the addition of reducing agents such as iron, tin, and antimony compounds^{39,40}) can the reduction of Cu(I) ions take place leading to the precipitation of metallic Cu nanoparticles. In the present case, conditions are not sufficiently reducing to give rise to Cu NP formation, even though it should be noted that prolonged heating of the film leads to a brownish color that could suggest the formation of some Cu NPs. However, no well-defined red color is obtained, possibly due to the coexistence of Cu(I) and Cu(0) species: As a result, the heating of the film should be stopped before the brownish color appears in order to characterize the film containing mainly Cu(I) species.

Follow-Up Assessment

This hands-on activity is easy to reproduce and is very engaging: It can be adopted in teaching laboratories for inorganic chemistry or physical chemistry for bachelor's students, as well as in a practical demonstration of redox reactions or in interdisciplinary activities about chemistry and art for high school students. The fast color change observed during the *in situ* reduction is well-suited for demonstrations at science fairs and other events.

We performed the activity twice with 20 high school teachers of different backgrounds (from chemistry to biology and forestry) during a refresher training course dedicated to the topic “Chemistry and Art”. Some of the participants had, by their own account, limited previous experience in basic lab skills (e.g., use of graduated pipettes), but this proved no obstacle in the successful completion of the activity due to the simplicity of the procedure. Before the hands-on activity, a 1 h seminar introduced the participants to light absorption induced by metal ions and nanoparticles (ligand field theory and plasmonics). The handout is included in Section S2. The participants showed a high degree of appreciation: some of them requested additional information in order to repeat the activity with their students. A survey of the participants' impressions about the activity was carried out (Section S3), and the results are reported in Section S4: The participants expressed a high degree of appreciation for the activity (9.1/10), and they found it didactically useful for the students (7.1/10). Moreover, 86% of the teachers expressed the intention to propose the activity to their high school students, but some of them noted that they lacked a spin-coater in their laboratories: As an alternative, a drop-casting deposition of the silica film was proposed, followed by a fast drying step in the oven (around 10 min) before the deposition of the metal salt solutions. It should be noted that square glass supports are

needed only if spin-coating is used for film deposition; if drop-casting is adopted for both depositions, rectangular glass slides can be adopted as well.

CONCLUSIONS

This hands-on activity can be adopted to introduced students of various levels (bachelor or high school) and backgrounds (from science to arts) to the chemistry of colors. This fast and easily reproducible experience also demonstrates redox reactions and the formation of metal nanoparticles, and it introduces students to the field of optical analysis.

The ancient procedure of stained-glass fabrication was mimicked by using a silica sol as a binder to attach the color-inducing metal ions to the glass plate by a low temperature, simple, and quick method. A hot-plate-activated reduction of the metal ions leads to the formation of Au and Ag nanoparticles, appreciable by the resulting plasmonic absorption bands. The optical analyses enabled correlation of the color of the colored glass plates with their chemical composition. The activity can be further extended by comparing the UV–vis spectra of lab-synthesized samples with those of commercial glass slides or even historical stained glass.

The participants greatly appreciated the engaging and tangible results, and they documented the entire experience with pictures and videos. The application-oriented nature of this hands-on activity and its ease of execution make it suitable for teaching laboratories as well as for public engagement activities.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.1c01141>.

Video displaying the *in situ* reduction procedure (MP4) Historical background, images of aluminum support for UV–vis measurements, photo of the silica film on a glass plate, wheel of colors, activity handouts, and evaluation survey on the activity and its results (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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