

Image Recording and Processing Chemical Synthesis: Method Description and Demonstration

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The reproducibility of chemical synthesis is of crucial importance for both chemical industry and research. The development of simple yet robust methods and algorithms that generate a set of data to help the community access their method's reproducibility is desirable. We report a method for obtaining, processing and analyzing recorded images, using scripting language Python 3, from any camera that allows continuous visualization and recording of a chemical synthesis.

The method can be used to assert synthesis reproducibility, to capture reaction events that might elude the naked eye and later be isolated in posterior reactions, to refine synthesis parameters such as temperature, microwave power and time and also to understand chemical reactions during synthesis. The method was exemplified with two microwave-assisted synthesis, zinc sulfide and silver sulfide, processed at our lab and applied to videos from other authors.

1. Introduction

Technological evolution has led many laboratories to embrace automation, owing to its capability to enable a much higher throughput in terms of synthesis completed in a shorter amount of time and in process control.^[1] Some aspects of this technological evolution led to a more secure work environment, enhanced data/features extraction and it has made the burden of repetitive work easier. Scientists can now observe and control their experiments from afar, using the internet,^[2] adding an extra layer of security, couple a series of measuring techniques in situ to optimize their experiments, particularly in the pharmaceutical industry where the product must be optimally pure.^[3,4]

The importance of chemical synthesis reproducibility is reflected on many aspects of the chemical industry and on research. As stated by Han et al., improving the reproducibility of synthesis and methods can save costs and time, with the given example of a planned large-scale chemical plant that, in case of a faulty chemical recipe can result in costly loss,^[5] and naturally improve the trust gained from peers and reliability of published articles. Normally, chemists rely on expensive spec-

trometers and apparatus to optically follow a reaction. A much cheaper alternative and versatile method is presented in this work, requiring only a simple inexpensive camera such as a mobile camera, coupled with a script to process the image data.

In terms of process control, in a dynamic system, data is acquired, quickly processed and new parameters are injected to correct or enhance the process^[6] or in a non-automated system, data is collected by the user and later analyzed to determine the new parameters. In the latter, which is usually the case for most chemists, it is fundamental to be able to acquire as much information from an experiment as possible.

Controlling and planning any given process such as a chemical synthesis can become a hurdle if the user is not aware of subtle changes in the reaction mixture. A simple detail might elude even the trained eye, or merely because the user was not present at the time of transformation, such a precipitation of a white substance from a milky suspension, among others. Therefore, it is important to monitor the color changes of reactions, for example: clock reactions,^[7] the precipitation of polymers or inorganic particles, ranging from nano to meso, the formation of organic molecules, etc. Further on, some examples of the application of the algorithm can be found on the Practical Examples section.

The continuous monitoring of the reaction by coupling a simple camera with a reaction setup, such as the one described by Bowman et al.,^[8] grants the user the possibility of observing the events happening during the synthesis. Specially if the setup is closed and it is not possible to directly observe it. Moreover, recording the images further enhances the analysis of said events. Processing the captured images using the feature extraction and selection method, which consists on transforming data and obtaining simpler and workable information, without compromising its integrity,^[9] can reveal fundamental details about the reaction. Any change in hue, brightness or contrast in a sequence of images, can be correlated to a phase change, precipitation, reaction, etc. A fully automated network of chemical robots was developed by Caramelli et al.^[10]

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to control an oscillating reaction and the script to reproduce their system was made available at <https://github.com/cro-ningp/NetworkedChemistryPlatform>. Not all syntheses require an automated response but would still benefit from a more quantitative imaging analysis. Several groups have presented a plethora of methods to acquire images and process them for fine tuning their experiments, as reviewed by Ley et al.,^[11] however, they do not provide a simple way for the general scientist to replicate the imaging and feature extraction process.

Here, a simple methodology for processing a sequence of images or a video is presented. This approach consists in the extracting the average red, green and blue pixel colors from each frame and plotting them in parallel with other observable features, such as temperature and pressure. The developed Python 3 script (Online Resource 1) and can be used across any system capable of running Python 3. The intent is to supply the scientist with a very simple method to record and extract quantitative information from their synthesis by giving them a python script that can be used without prior programming knowledge. It is not an upgrade to the already existing and excellent methods but it serves as a complement to be used on the daily basis.

2. Results and Discussion

2.1. Method Description and Application Examples

2.1.1. Recording Images of the Reaction Vessel Over Time

Both a video and a set of images can be processed by the Python script. Ideally, there should be no variation of ambient light, so that the observed changes are not influenced by external light fluctuations. If there are any fluctuations, consider correcting the brightness with the parameter `WhiteXY` and `BlackXY`, which should correspond to the areas of the video where the maximum white and black, respectively, are considered. Lock the camera's contrast, brightness and focal length to minimize incorrect color changes.

2.1.2. Converting the Video or Images to Color Data

Run the functions `"grab_images"` or `"grab_video"` and attribute to a variable which will store the color data. In the case of using images as the color data source, format the name of each image as `"name_HH_MM_SS"`, where name can be anything as long as it does not include the symbol `"_"`, HH are the hours, MM the minutes and SS the seconds.

2.1.3. Acquire the Temperature Data (Optional)

Attribute to a variable the time and temperature profiles as suggested in the Python 3 script from the supplementary material. If the user has an Anton Paar Monowave 400 micro-

wave reactor, the csv file outputted by the system can be automatically processed by the `"grab_data"` function.

If the user has another source of parameters such as for the temperature or pressure, this data should be saved in a csv file with the first line with the columns [Time, Step Time, Temp 1, Temp 2, Pressure, Power, Stirrer Speed, Fan Level, Step No.]. The following line should contain the units for each parameter. Time and Step Time should be in seconds and the values can be of float type (contain decimals). An example file (Online Resource 2) is available in the supplementary material. The function to extract data from the csv file `"grab_data"` has a second argument to discern if the input's origin is a file from Anton Paar's microwave reactor and is set to `"False"`; not from Anton Paar as default.

2.1.4. Plotting the Data (Optional) and Color Data

Insert the data and color data variables into the function `"plot_colors"`, with the parameter `"T"` equal to 1 to show the temperature profile along with the red, green and blue pixel color profiles.

The following examples illustrate the expected data that can be extracted from a set of images or a video. The correlation between color transitions and other measurements presented below are for demonstration purposes and should be regarded as such. The user should carefully evaluate the composition of the synthesized substance immediately before and after a color transition to prove the correlation the color transitions and the phase or composition changes.

Our group develops nanomaterials and one of the main concerns is synthesis control. The script described in this work is routinely used for the latter. An example with a video (Online Resource 3) and image files (Online Resource 4), taken from a Anton Paar Monowave 400 microwave reactor is available, along with the Python 3 script with the necessary functions.

The two color and temperature profiles are examples of two synthesis done by our group, which consist in the formation of zinc sulfide and silver sulfide in a microwave reactor, as can be seen in Figure 1.

Some of the events that occur during the zinc sulfide synthesis, confirmed and reported elsewhere,^[12] are not easily recognizable to the naked eye. In Figure 1 (A1) a gel can be seen forming after a mixture of zinc acetate and a thiol is heated, followed by its dissolution. At around 275 °C, in (A2) a slight color variation is observed, signaling the formation of ZnS, as the XRD pattern indicates. After 2 to 3 minutes, a small bump in the color profile (A3) reveals that the ZnS precipitates. Notice the difference between the second frame from (A1) and the frame from (A2) are practically equal to the naked eye but the XRD patterns show that the substances are completely different. The user, at the first glance, would assume that ZnS would only be formed in (A3).

As for silver sulfide synthesis, in Figure 1 (B1), the silver salt quickly dissolves, forming a red complex with the thiol. This is followed by a color change to yellow, then to red again (B2) and finally the black silver sulfide is formed (B3), confirmed by

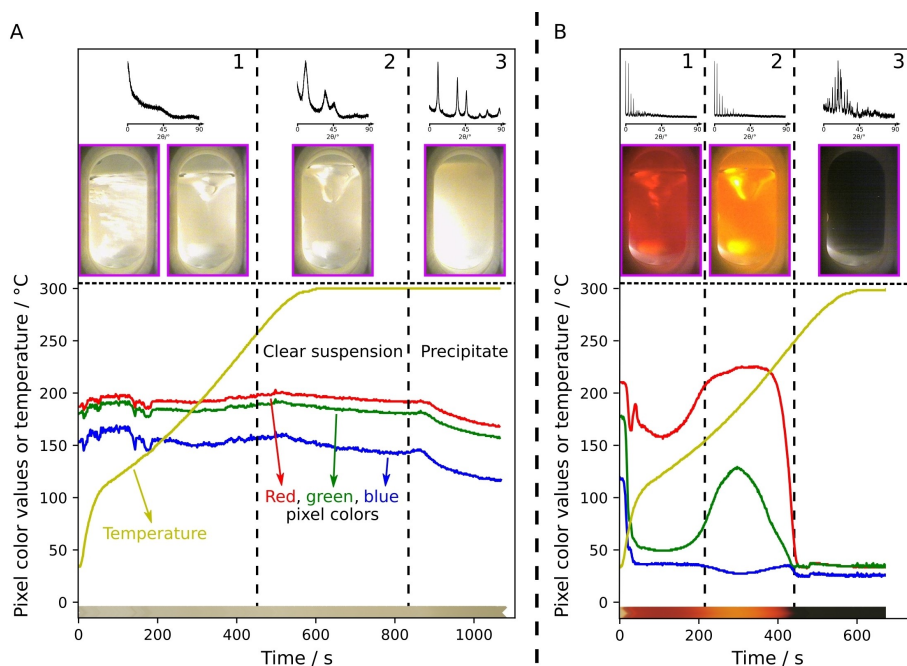


Figure 1. Red, green and blue color profiles over time of zinc sulfide (A) and silver sulfide (B) microwave-assisted synthesis. Each graphic is divided into three parts and each part is composed of a XRD pattern of the substance taken in the corresponding time range, a picture of the reaction vessel and the temperature and red, green and blue pixel color values.

the XRD. The plethora of color changes can be clearly separated and the products isolated.

The script can also process a video. Figure 2 shows two plots, one recorded from the microwave reactor's internal camera (Online Resource 4) and another from a mobile's camera (Online Resource 3) which recorded the microwave reactor's screen that was showing images from the same reaction captured by the internal camera.

The difference of data resolution is due to the difference in the frame rate capabilities. The mobile camera recorded at 30 frames per seconds, while the reactor's camera only recorded at 1 frame per second. Recording synthesis in this manner and plotting the color profiles can also be used to check the reproducibility of a synthesis, not only in a microwave equipment that has an internal camera but any other synthesis that

can be assessed by monitoring it with a video camera. In Figure 3, lies two synthesis of silver sulfide that were done in the same manner.

The similarity between both color profiles shows that the microwave-assisted synthesis of silver sulfide is reproducible. The initial part of the synthesis does not always begin at the same temperature, given that room temperature varies. After some time, the automated temperature control stabilizes the temperature profile and the transformation from a red colored solution to a black suspension happens on the same time frame, when comparing both syntheses. The difference in color intensity is due to the automatic contrast setting from the microwave reactor and not due to actual differences in illumination.

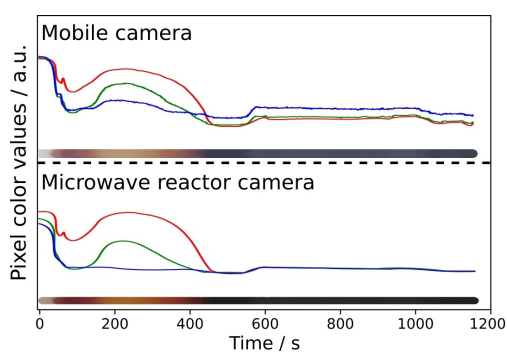


Figure 2. Red, green and blue color profiles over time of silver sulfide synthesis recorded with either a mobile camera or the microwave reactor's camera.

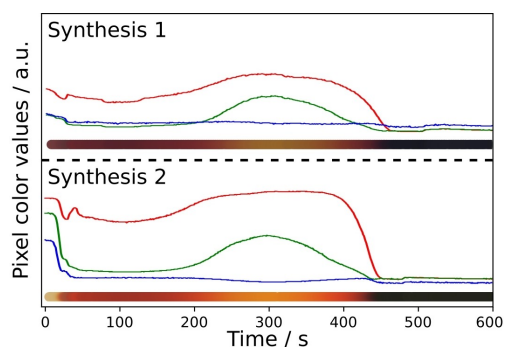


Figure 3. Red, green and blue color profiles over time of two silver sulfide synthesis.

2.2. Limitations

Depending on the video camera's sensitivity, if there is excessive or too little illumination, the contrast will be too low and any color change will not be noticeable. There are multiple methods to enhance the contrast of an image or to convert to other color spaces such as HSV.^[13] The user should keep the original recording to verify if a given color change is in fact due to physico-chemical change. A white light lamp source is advised but it is also possible to use a monochromatic light source in some cases, at the user's discretion. In terms of statistics, recording two synthesis replicas should yield similar color profiles, unless the light setup differs between experiments. Such is the case of the experiments shown in Figure 3. Synthesis 1 had a different contrast setting but the color transformations can still be evaluated in synchronization with Synthesis 2.

2.3. Examples from Other Sources

To assure that the script presented in this work can be broadly used for any kind of image sequence or video, examples taken from an article and two YouTube videos were processed using this work's script and the results are shown in the following sections. Any analysis described in this work, regarding the examples below are not from the original content authors and should be regarded solely as hypothetical.

2.4. Article

Bowman et al. recorded a series of microwave-assisted synthesis and one of them was a simple reduction of palladium acetate to metallic palladium. They observed that after irradiating the orange solution with microwave radiation, metallic palladium precipitated after some time. However, after around 15 seconds, severe arcing developed and they were forced to cut the power from the microwave source, to prevent further damage to the system. Using the script to extract the color profile from video 12, available in the supplementary material from reference,^[8] Figure 4 was obtained.

In this example, no significant detail is obtainable but if the color profile data was inputted to a system that would automatically detect a sudden color spike to white and cut the microwave power, it would prevent a catastrophic synthesis; the temperatures reached in arcing.

2.5. YouTube videos

The examples given above were all from microwave-assisted nanoparticle synthesis where the temperature and pressure data are easily synchronized with the images, since both the images and the data are outputted by the same system. The script will also output a trustworthy color profile for other types of synthesis or reactions done in a conventional hotplate, as

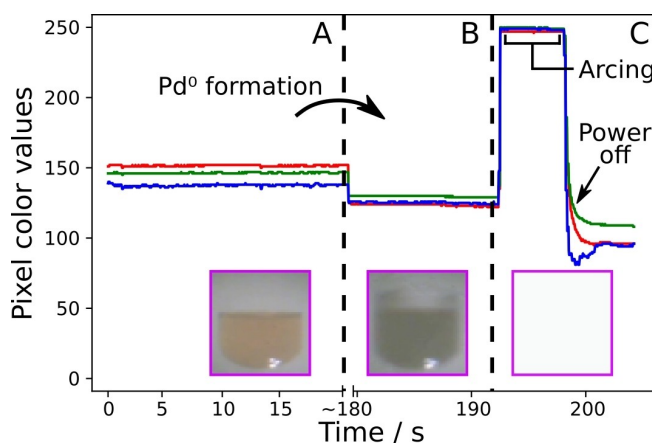


Figure 4. Red, green and blue color profiles over time of palladium acetate (A) reduction to metallic palladium (B) and arc formation (C). From (A) to (B) there is a time skip of around 3.5 minutes. In (C), the microwave irradiation was turned off after ~5 seconds of arcing (Power off). The insets show a representative frame from the video of the corresponding section A, B or C.

long as the user is able to match the recorded images with the other data, for instance synthesis temperature and time. An example that does not involve heating a mixture is the Briggs-Rauscher oscillating reaction^[14] that has countless videos on the internet, demonstrating the oscillating clock effect. Two videos were taken from YouTube, to serve as target examples for the script. In Video 1,^[15] it is possible to see the clock reaction with up to six cycles of transitions between a transparent yellow and a very dark blue suspension. In Video 2,^[16] four cycles are observed before the author stopped recording. Figure 5 contains the extracted color data of each video, respectively denoted as Video 1 and Video 2. For each video, segments A1 and A2 contain the average red pixel color from each video's frame, accompanied by an inset showing the region of the video that was used in the script. Segment B1 and B2 is a plot of the yellow and dark cycles that were fitted with a simple power function from the data in segment A1 and A2, respectively.

The data extracted from Figure 5 A1 and A2, presented in B1 and B2, could be used to study the kinetics of the clock reaction, for example to optimize the duration of each cycle by changing the reactants quantities or addition speed.

In a pedagogical article published in 2018, the authors suggest a practical class to demonstrate the clock effect on a paper substrate instead of in solution.^[10] Given that nowadays access to cameras is easy, instead of relying on chronometers, students could record the reaction and later analyze with the script presented in this work.

3. Conclusions

A Python script was developed and its application in the plotting of color features from camera or video images of a chemical synthesis was demonstrated. The resulting color profiles enable to prove reproducibility, detect phase changes,

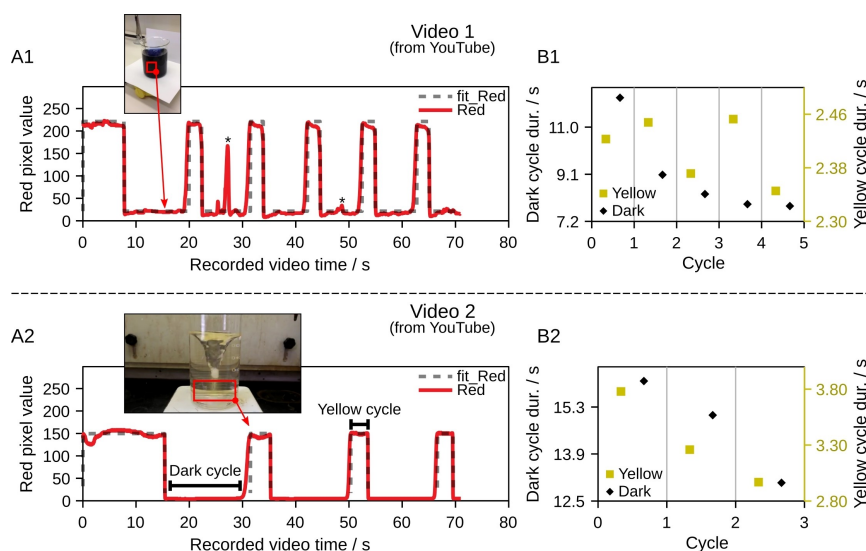


Figure 5. A1 and A2 – Plots of the average red pixel color with insets from a video frame, containing a rectangle that indicates where the pixel color data was retrieved from. B1 and B2 – plots of the duration of dark and yellow cycles. (A1, B1) and (A2, B2) were obtained from YouTube's Video 1 and Video 2, respectively. The asterisk (*) denotes light artifacts during the video recording.

or intermediate reactions that might be not visible to the naked eye. This is an important aspect for all chemical synthesis, including but not limited to nanoparticles, polymers and organic and inorganic compounds, where a single failure can cost several hours of work and reagents. It was also shown that the script can be used generically in any image sequence or video. The Python script is supplied and can be easily used in any compatible operation system.

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Conflict of Interest

The authors declare no conflict of interest.

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