Preliminary Results: Directed Deposition of Photovoltaic Inks using Clay Particles

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Reference XRD Patterns

In the later stages of the investigation, it is expected that the diffraction patterns of the sedimented thin films will contain peaks and features of both the sedimented clay and the CIGS particles. To identify which peaks belong to which constituents, it was necessary to perform several reference experiments: one for each powdered clay, one for the aluminium sample holder, and another for the kapton lid. These reference samples will also aid in establishing the changes of the thin film structure during and post-sedimentation, as well as ascertaining the arrangement of the CIGS particles in the thin film.

In order to obtain these reference datasets, the Brucker D8 Advance XRD machine was used. Each of the three powdered clay samples were exposed to the x-ray source for an extended period of time (i.e. at least 10 hours in each case), which gave rise to an appreciable signal-to-noise ratio, suitable for use as a reference dataset.

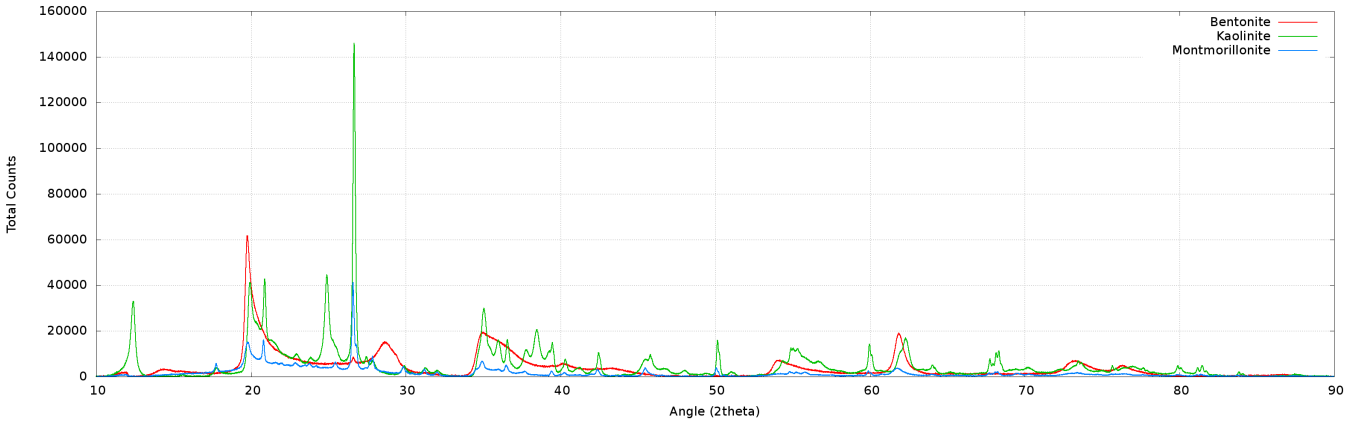

Figure 1: *Reference diffraction patterns for powdered clays: Kaolinite, Bentonite, & Montmorillonite.*

Time Dependent XRD

After the reference samples were obtained, a time-dependant sedimentation experiment was conducted. The aim of this experiment was to obtain a series of time-dependant spectra throughout the consolidation step for each of the investigated clays. To date, only montmorillonite has been tested in this manner, with plans to conduct the same method on the remaining two clays in the following week. The full time-dependant datasets for each clay will consist of four diffraction measurements taken over the period of two days.

The time-dependant samples are prepared by formulating a suspension of toluene and the chosen clay. This suspension is then placed into a custom sample holder that is specifically engineered to allow the sedimentation to take place in the same environment as the final x-ray diffraction measurement. Once the suspension is deposited into the sample holder, it is placed in a fume cupboard for a period of 96 hours. At the conclusion of the initial sedimentation, the excess toluene is removed and a kapton lid is placed on top of the sample holder. The kapton has a high x-ray transmittance and prevents the remaining toluene from evaporating away, allowing the consolidation step to continue unhindered. Time-dependant XRD can then be commenced on the samples.

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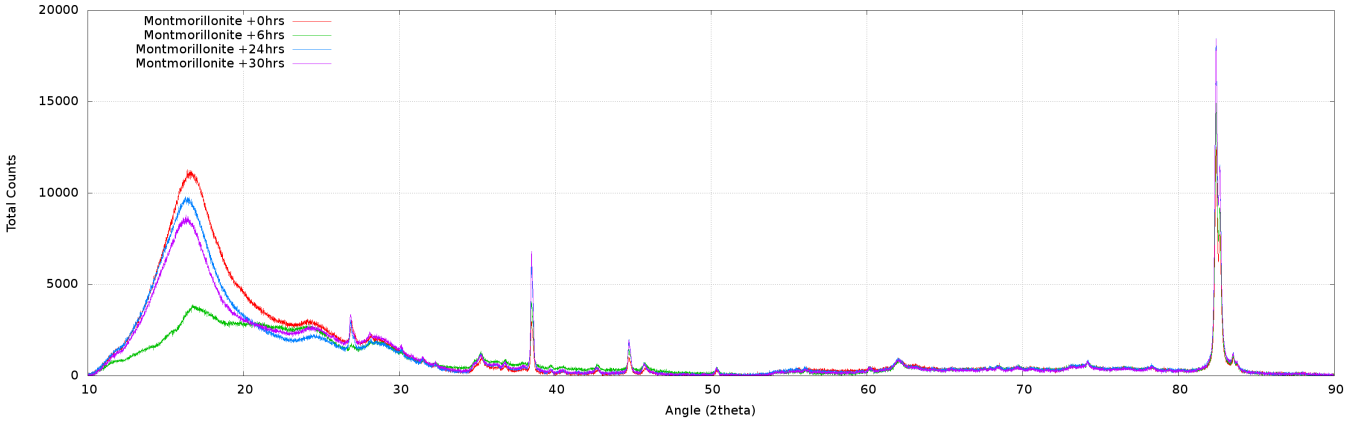

Figure 2: *Time-dependant set of diffraction patterns for Montmorillonite.*

Initial Analysis & Project Direction

The clay reference datasets have been corrected for background radiation and overlaid in figure 1. At first glance, it appears that there are several common peaks between the clays. This was expected as the clays contain similar constituents and structure. For example, kaolinite and montmorillonite are both phyllosilicates, which means that they contain parallel sheets of silicate tetrahedra. Furthermore, montmorillonite is the bulk material that makes up bentonite. Whilst kaolinite and montmorillonite have very defined, high quality peaks, bentonite consists of nanoparticles that have the undesired effect of peak broadening. This is a possible reason why the montmorillonite peaks in the bentonite data set are not visible in the diffraction pattern.

The time-dependant datasets for montmorillonite behaved unexpectedly. As shown in figure 2, there appears to be a large broad peak in the lower angles, centred on 16.4^o. It has been suggested that this is due to long-range disorder in the sedimentation micro-layers due to the presence of toluene in the void space. Another possible reason is strain occurring in the kapton layer above the sample. The kapton lid is designed to be airtight to prevent the evaporation of the remaining toluene, however this has the undesired effect of causing the kapton layer to bow outwards due to increased air pressure induced inside the sample environment when the lid is pressed down. Upon cross-referencing the toluene and kapton reference datasets with the time-dependant datasets, the long-range disorder hypothesis seems more plausible, but a conclusion cannot be finalised until the bentonite and kaolinite samples have been analysed. It may also be necessary to repeat the montmorillonite time-dependant experiment due to a disturbance of the sample between the +6hrs and +24hrs measurements, which occurred due to human error. This caused a disagreement between the first two measurements and the final two measurements. As the clay progresses through the consolidation stage, it is predicted that diffraction peaks corresponding to the preferentially orientated lattice planes will increase in intensity, whereas the non-preferential peaks will decrease in intensity. The montmorillonite time-dependant data does not seem to show this, which may be because of the aforementioned disturbance to the sedimentation layer.

The more immediate concerns of the project are to collect the remaining time-dependant XRD measurements and to repeat the montmorillonite time-dependant experiment. Furthermore, the excess toluene in each case will be allowed to evaporate away to leave dry sediment clay samples. XRD analysis will be performed on these dry samples to ascertain the final state of the post-sediment clay. The data from all of these experiments will be analysed thoroughly and a clay will be chosen with which to conduct CIGS-doped sedimentation in the main stages of the experimentation.