MICROSCOPICAL EXPLORATION THIRTEEN

An investigation into the effect of the addition of caffeine on the crystal structures of commercially available aspirin brands at low concentration following recrystallization from ethyl ethanoate solution

Introduction

The solubility of acetylsalicylic acid (aspirin) decreases in the order of ethanol, ethyl ethanoate, carbon tetrachloride, xylene, and water. Due to the easy availability of, the relatively high solubility of aspirin in and the low boiling point/high volatility of ethyl ethanoate, this is the preferred solvent for this investigation. For the investigation six different brands of 300mg aspirin tablets were chosen. The procedure detailed below was carried out separately for each brand and follows on from Microscopical Explorations 11 and 12 which were conducted at higher aspirin concentration, without and with the addition of caffeine respectively.

Aim

To identify by conventional and polarised optical microscopy any variations in the crystal structures of commercially available aspirin brands with added caffeine when recrystallized from solution in ethyl ethanoate.

Applicability

The procedure is applicable to any commercially available aspirin tablets.

Equipment

Small glass bottles with lids.
Filter funnel and filter paper
Mortar & pestle or two teaspoons (for crushing tablets)
Vickers M10A optical microscope equipped with incandescent filament bulb white light illumination and sub-stage condenser.
Polarising filter and analyser
Digital eyepiece camera (Brunel Microscopes Ltd. Eyecam) to replace the x10 microscope eyepiece.
Microscope slides
Dropping pipette

Materials/Reagents

Branded aspirin tablets: Anadin Original, Aspar brand, Boots brand, Morrisons brand, Tesco brand, Wilko brand. It should be noted that Anadin Original tablets already contain approx. 4.4%w/w caffeine.
Ethyl ethanoate (acetone free nail polish remover).

Procedure

A tablet containing a total of **300**mg of aspirin was finely crushed between two teaspoons and the resulting powder was well mixed with 20cm^3 of ethyl ethanoate at room temperature and allowed to equilibrate for 30 mins. Assuming complete dissolution of the aspirin this yields an aspirin solution of **15** mg/cm^3. To this solution was added, with mixing, one finely crushed 50mg caffeine tablet. The solution was allowed to equilibrate once again and then clarified by filtration through filter paper circles cut from coffee filters Two drops of the clear solution were applied to the centre of a glass microscope slide and allowed to evaporate slowly overnight at room temperature. The crystals of aspirin/caffeine thus formed on the slide were observed microscopically and photographed for each brand as follows:

Image 1 X4 objective with transmitted white light illumination and sub-stage condenser only.

Image 2 X4 objective with transmitted white light illumination, polarizing filter immediately above the sub-stage condenser and analyser between the microscope objective turret and the digital eyepiece camera.

Observations

Anadin Original

Image 1

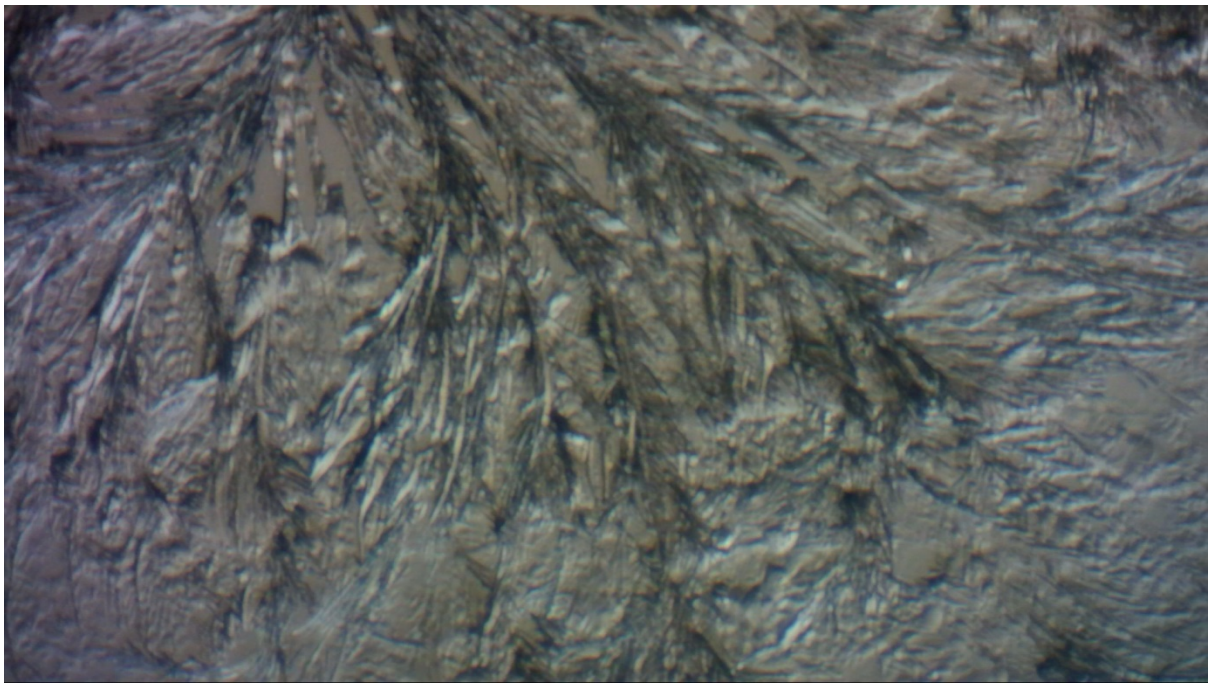
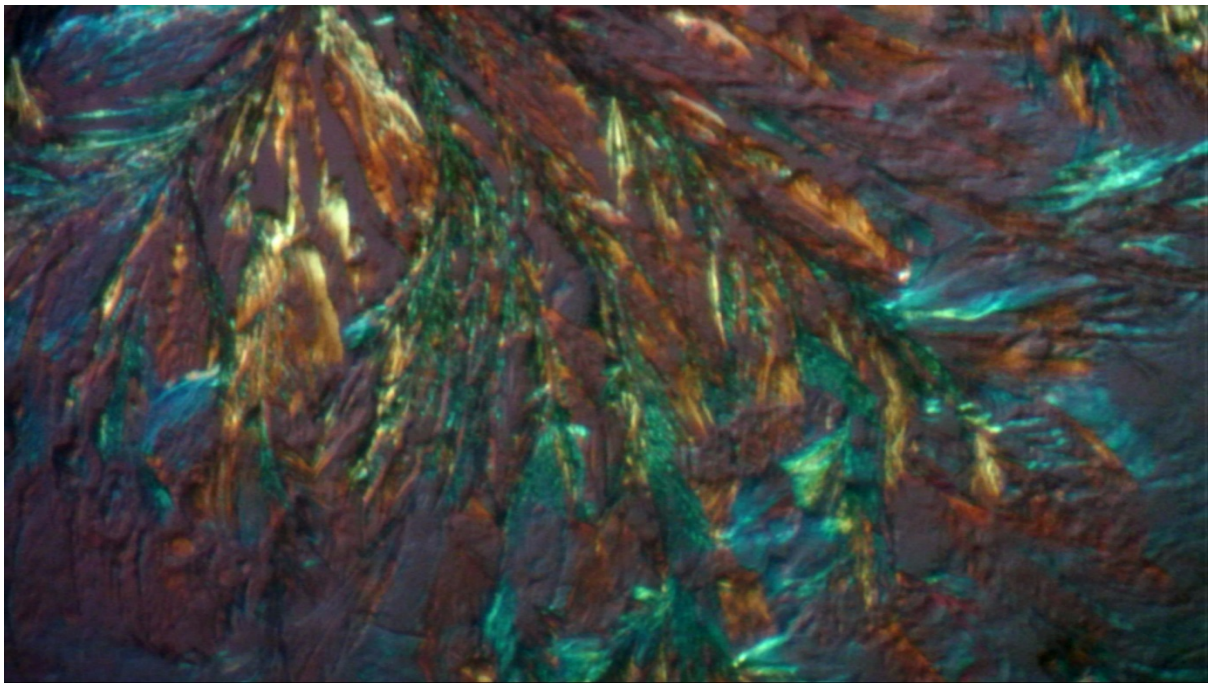


Image 2



Aspar Brand

Image 1

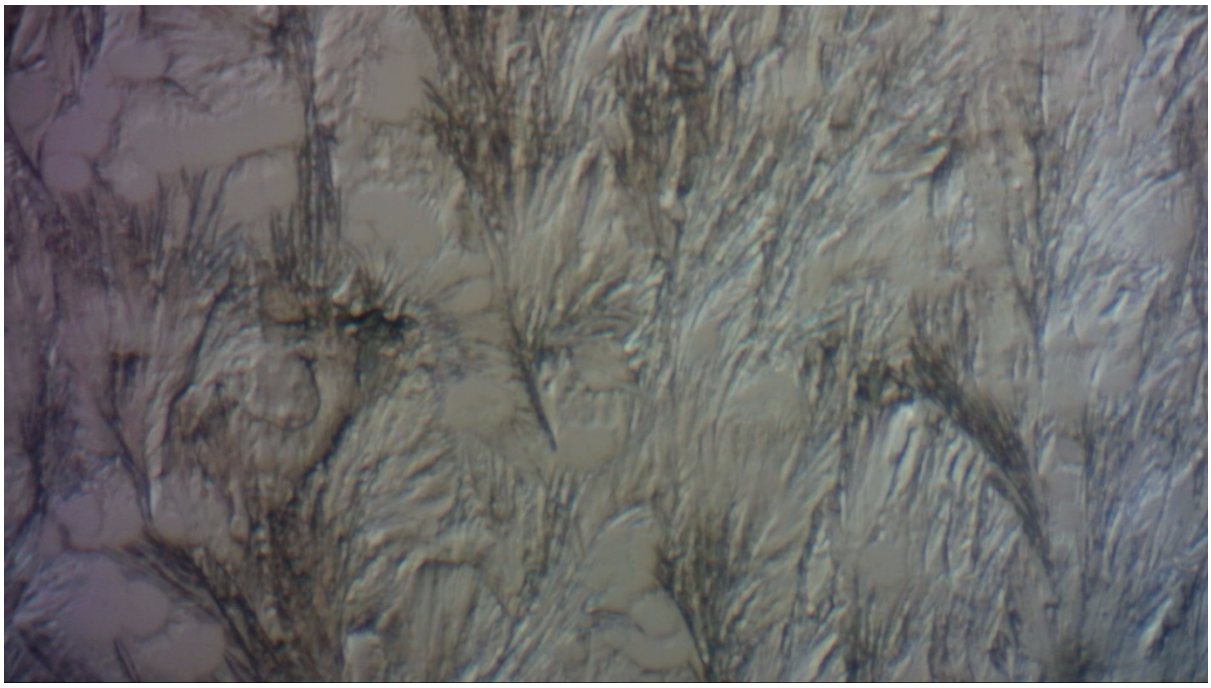
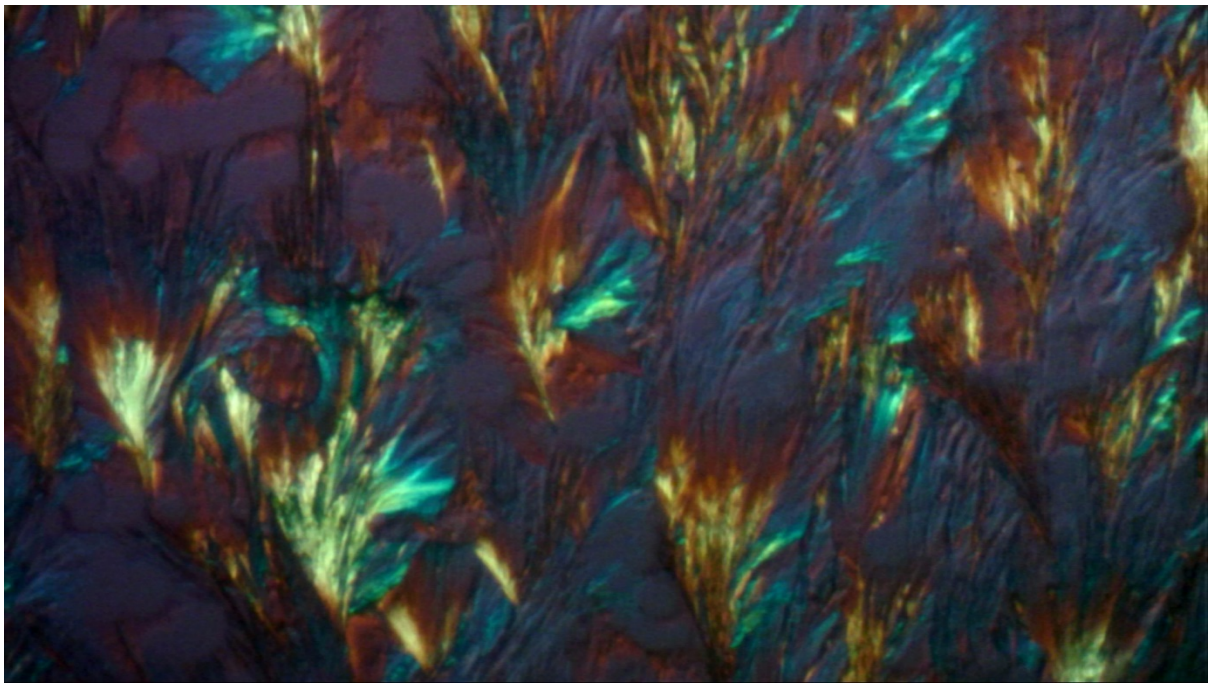


Image 2



Boots Brand

Image 1

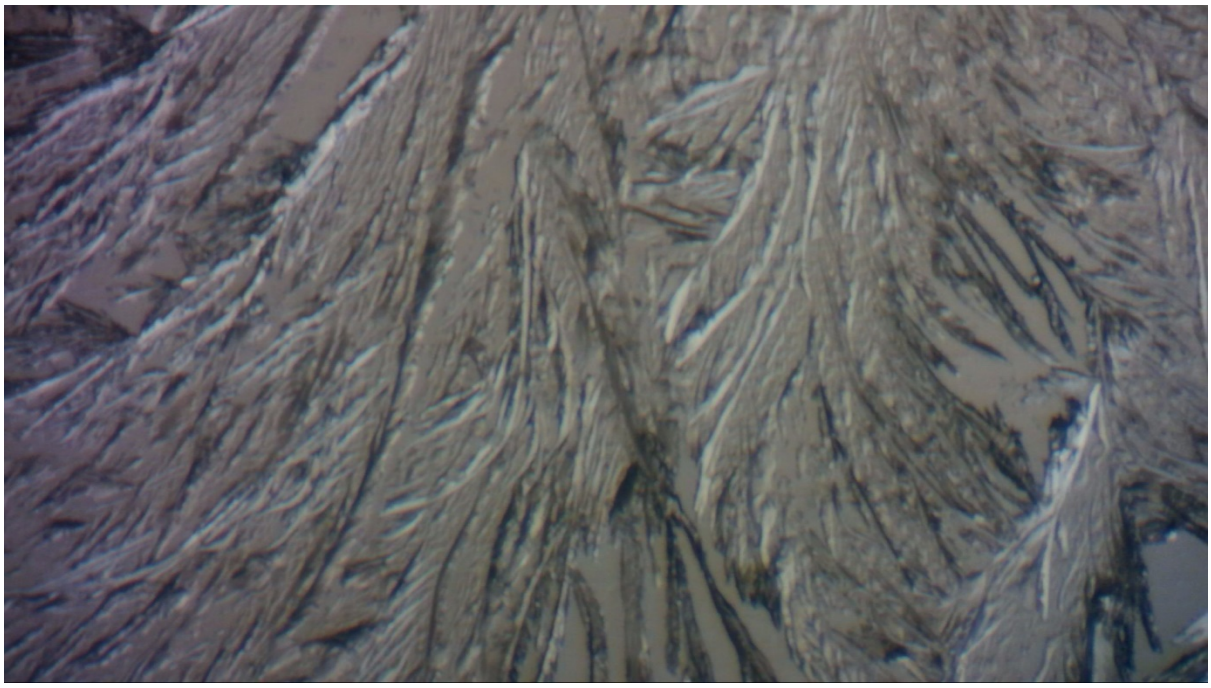
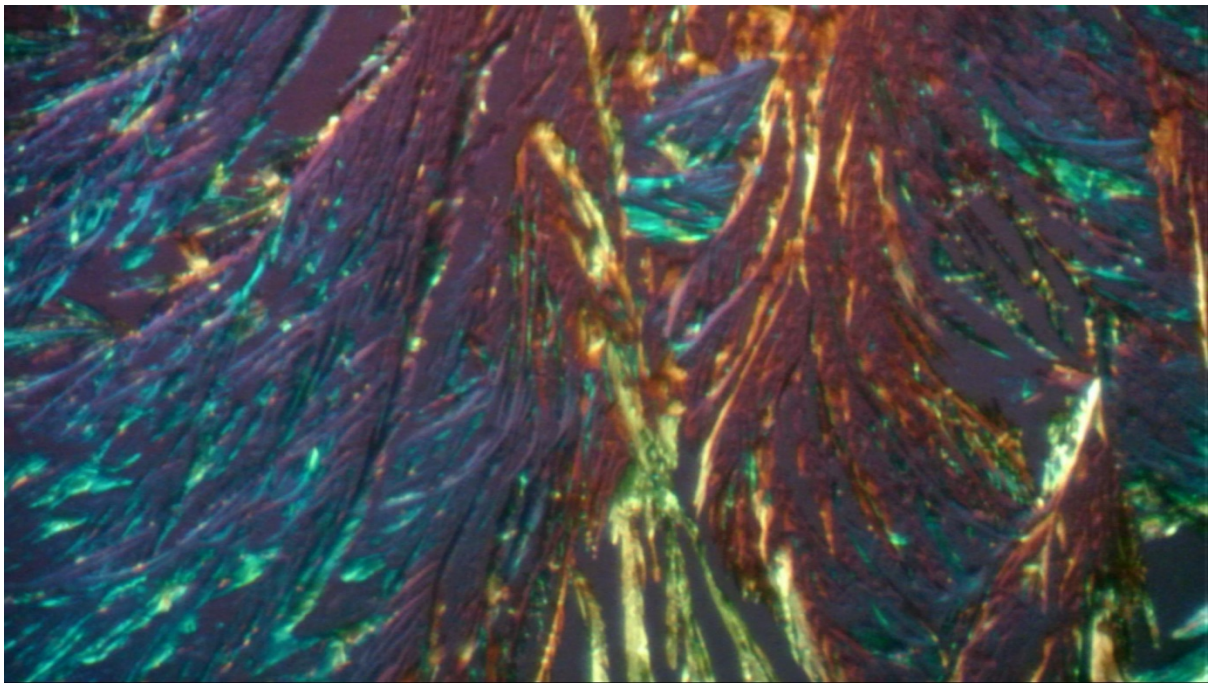


Image 2



Morrisons Brand

Image 1

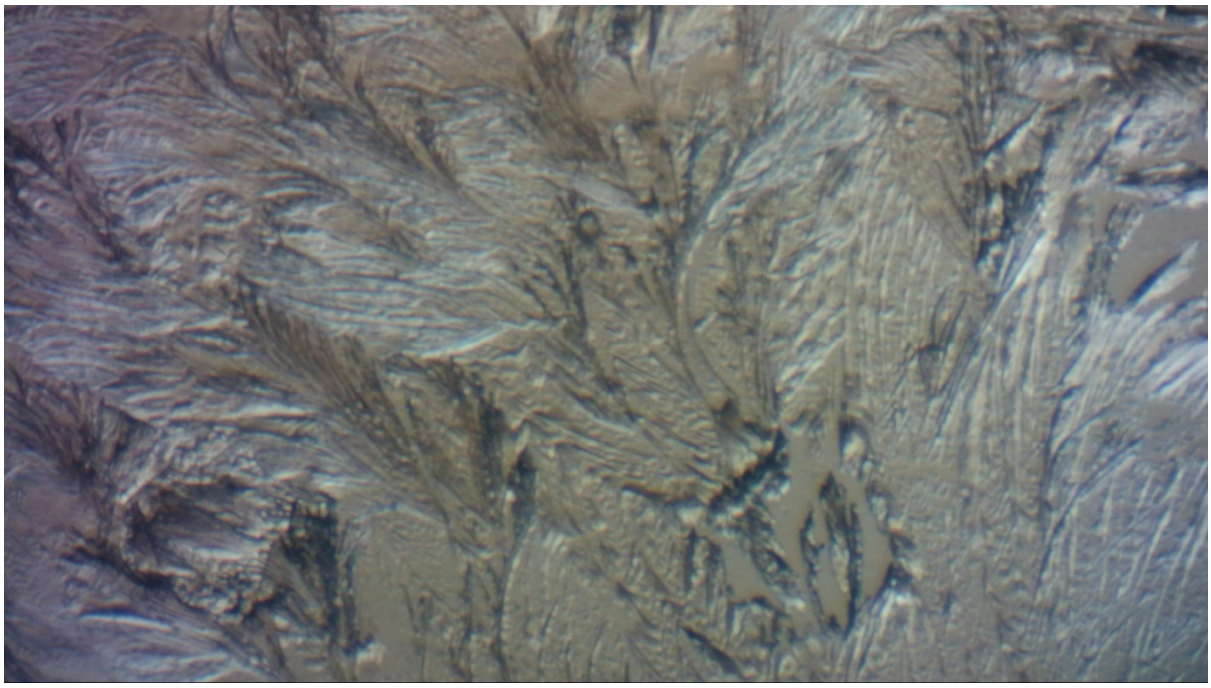
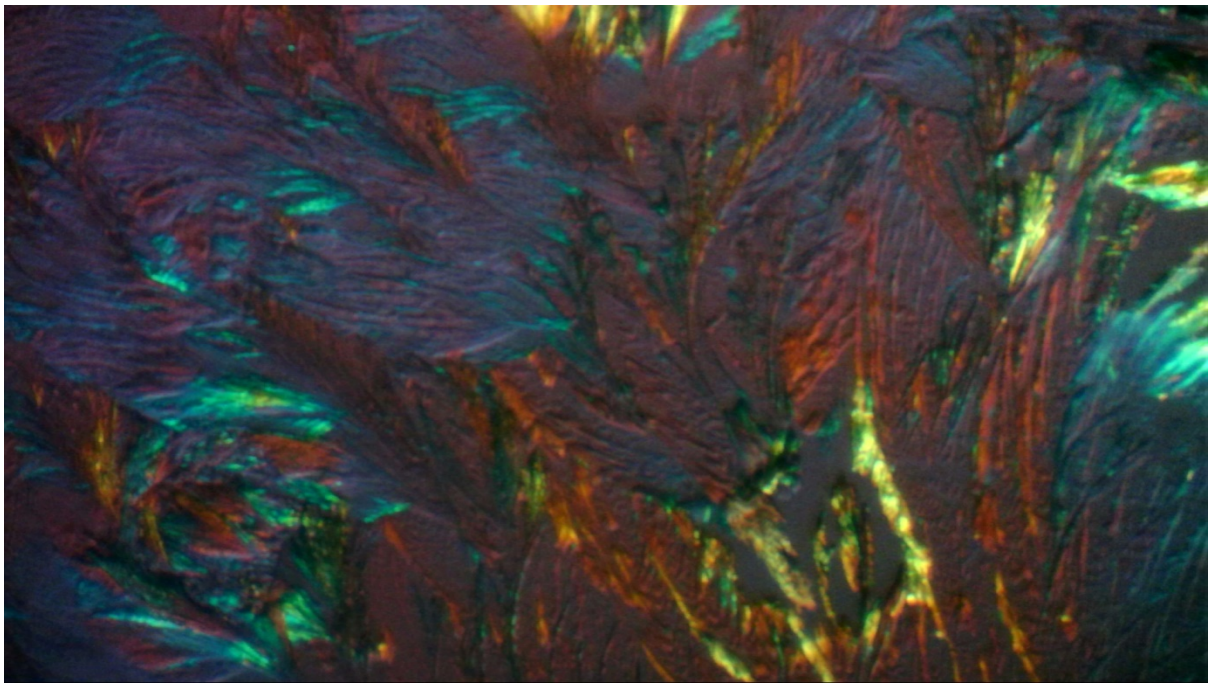


Image 2



Tesco Brand

Image 1

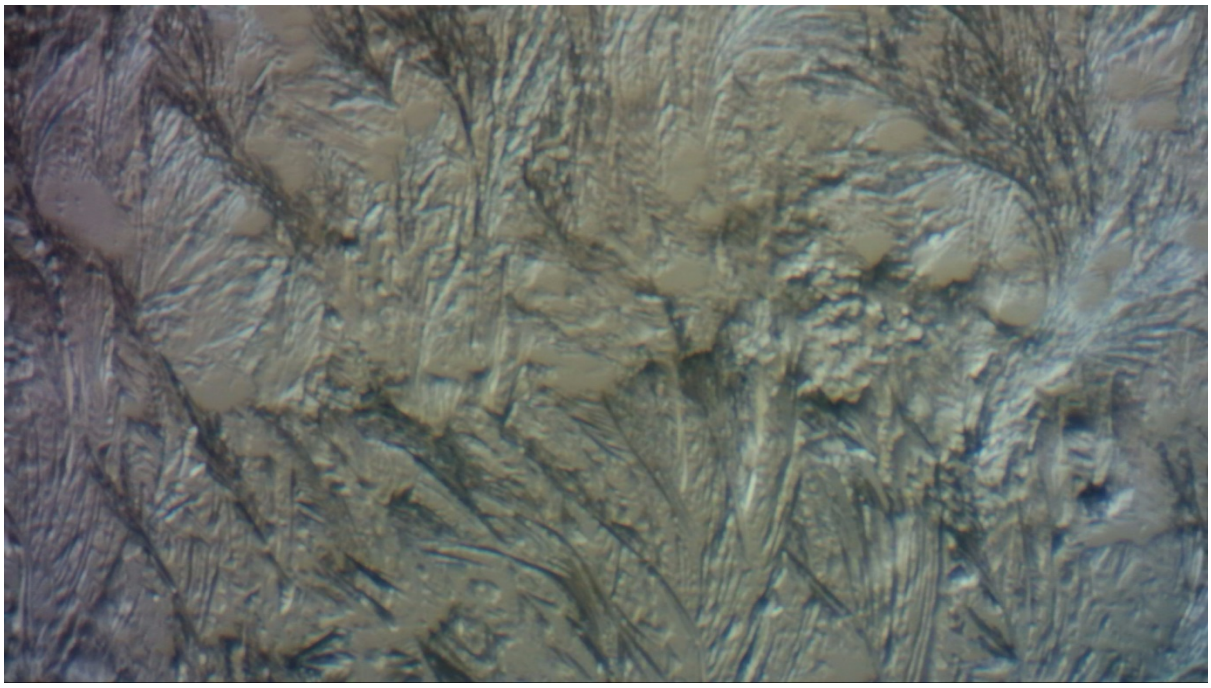
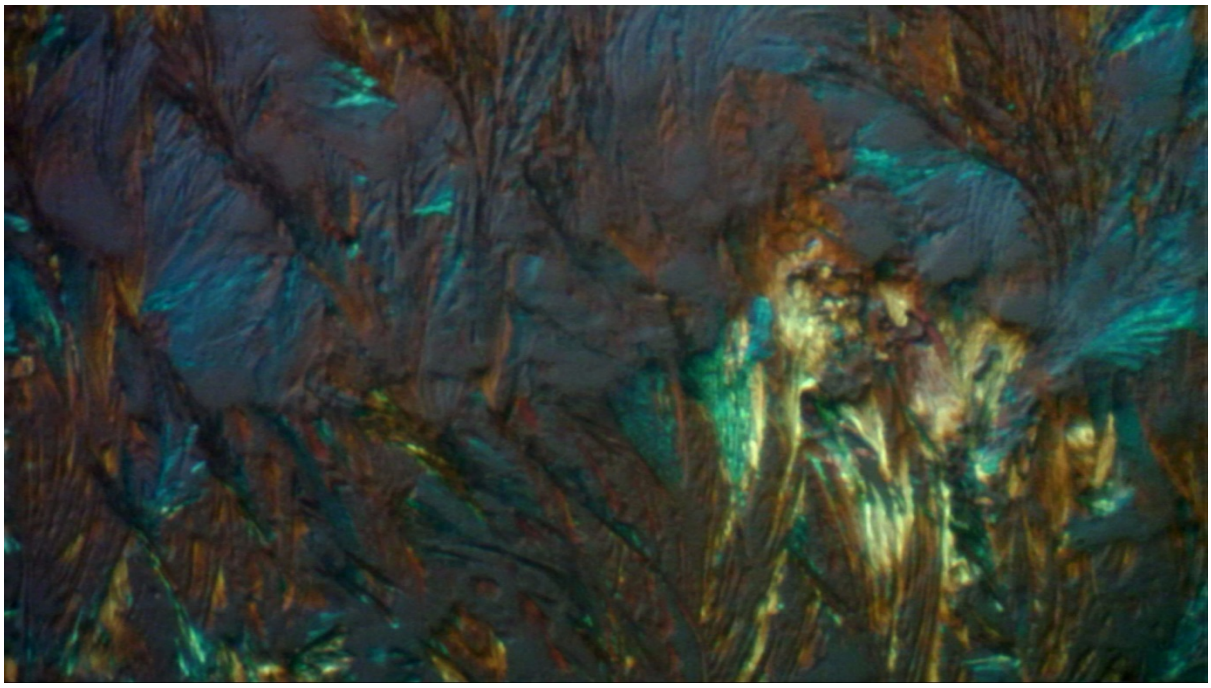


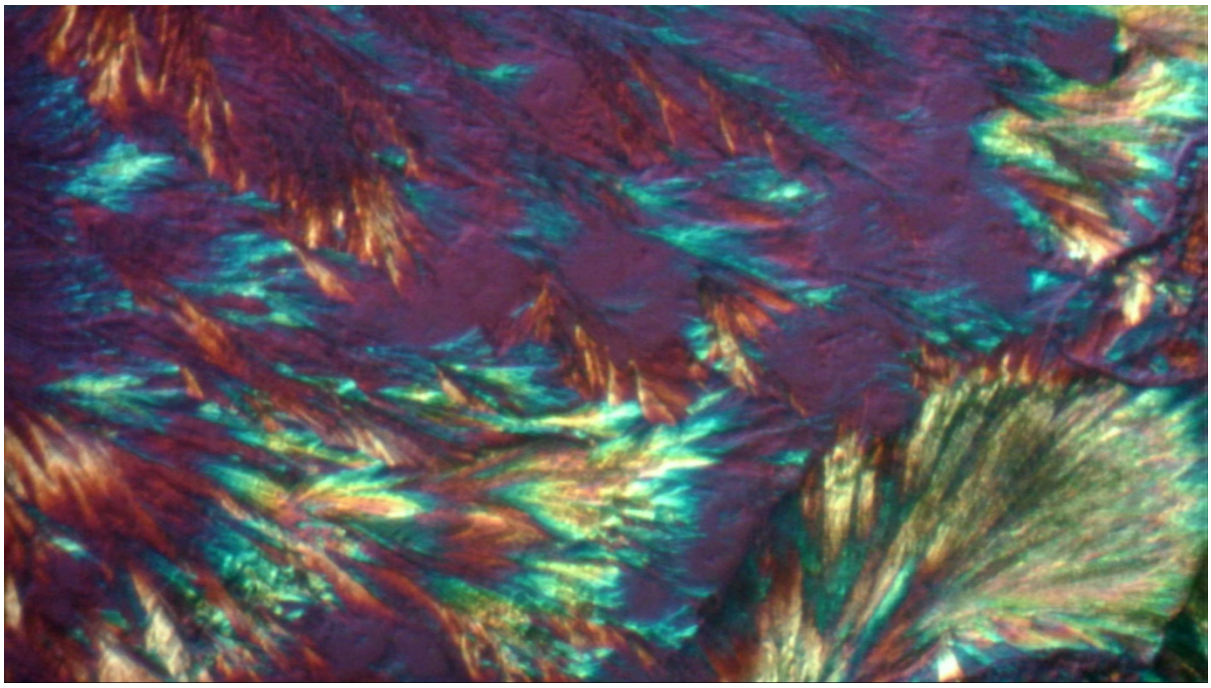
Image 2



Wilko Brand + caffeine

Image 1

Image 2



Conclusions

As also observed in Microscopical Exploration 12, at higher aspirin concentration, the addition of caffeine to more dilute solutions of branded aspirin in this investigation also results in the complex co-crystallization of the two compounds and the feather-like crystal matrix observed.

References

CRC Handbook of Chemistry and Physics CD ROM 2005.

CRC Handbook of Chemistry and Physics 58th Edition 1977-1978.

Electronic Supplementary Material (ESI) for CrystEngComm. © The Royal Society of Chemistry 2015.

Once again, interpret these abstract pictures as you will, but as we say here in Cumbria:

'Ave a go yersel'!

James Stewart

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