	Experiment Title: Study of the 3-d long range ordering in dispersions of novel colloidal cubes	**Experiment number:** 26-02-525
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Shifts: 9	**Local contact(s);** Dr. Guiseppe Portale	
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Report: (max. 2 pages)

In the performed experiment we have studied a long-range periodic order formed in dispercions of hematite (β-FeOOH) colloidal cubes [1-2] by the method of microradian x-ray diffraction (μXRD) [3]. A wide range of samples was synthesized for this experiment. Sizes of cubes varied from 300 nm to 1000 nm, some of them were coated with silica and also different solvents (pH) were tested in dispersions. For example in the Figure 1 we presented TEM pictures of hematite cubes. Pure hematite cubes (Fig 1a) have an edge length 270 nm and silica coated cubes (Fig 1b) have an edge length 340 nm correspondingly.

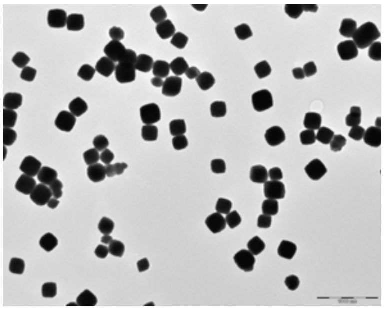
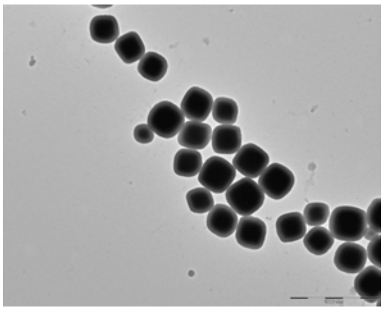


Fig 1a

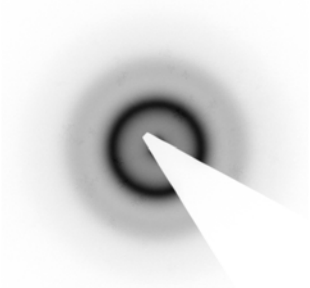


1b

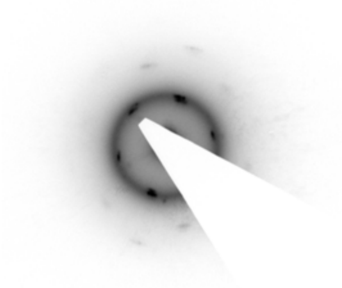
TEM micrograph of the hematite cubes with edge length 270 nm 1a) and hematite cubes coated with silica edge length 340 nm 1b)

Dispersions were placed in vertically-stored containers for sedimentation. We did study of the ordering formed at different high in the cappilaries by the vertical scan, using a motor controlled translation-rotation goniometer stage. Selected, highly ordered, areas in samples were examined by rotation scan to reveal a 3-D crystalline structure. Similar procedure was applied for samples immersed in the external magnetic field.
Here we present selected examples from our experiment without going deep into detailed analysis of structures in dispersions which would require detailed quantitative analysis of translational-rotational scans.

Fig 2a

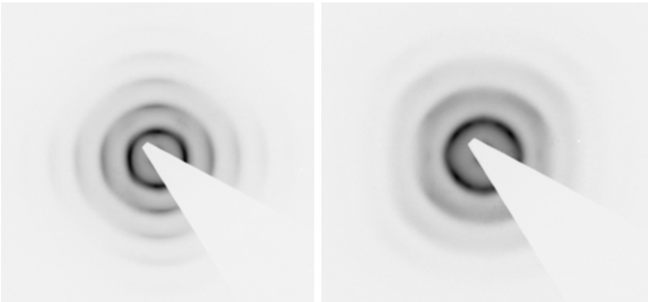


2b



μXRD patterns of the hematite cubes (270 nm) 1a) and silica coated (340 nm 1b)

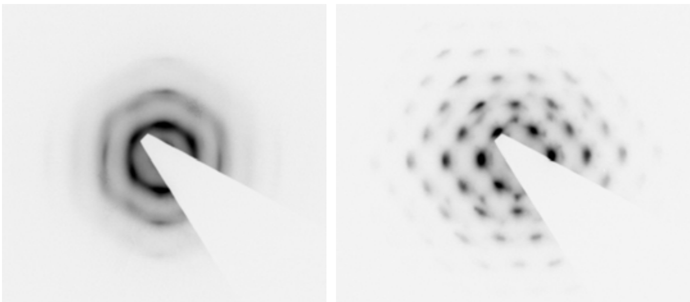
On the figure 2 we present characteristic μXRD patterns obtained with the same material as was shown on figure 1. Dispersion of hematite cubes in water (Fig. 2a) posses broad peaks q_1 = 0.0198 nm^{-1} ($2\pi/q_1$=317 nm) from the positional ordering in the sediment while the long range orientational ordering is not present.
Dispersion of Silica coated cubes in ethanol (Fig. 2b) also has a broad peak at q_1 = 0.0157 nm^{-1} ($2\pi/q_1$=400 nm) and on the top of that we have sharp peaks originating from 3-D crystalline ordering in the sample.
Another prominent example is hematite cubes with an edge length of 500 nm in water, Figure 3a. Here we also have preferable orientation in the sample which change their direction upon application of external magnetic field figure 3b. The magnetic field was horizontal in our experiments.



μXRD patterns of the hematite cubes with edge length 500 nm 1a); the external magnetic field of 25.5 mT applied in horizontal direction

Fig 3a 3b

Diffraction patterns of the same cubes coated with silica layer (thickness of the layer is 70 nm) and dispersed in ethanol presented at Fig 4a. The sediment has strong 3-d positional and orientational ordering. When magnetic field applied, the particles rearrange and possess strong 3-d crystalline ordering.



μXRD patterns of the hematite cubes coated with silica (640 nm) 1a); an external magnetic field of 25.5 mT applied in horizontal direction

Fig 4a 4b

To summarize we highlight that μXRD experiment revealed a wide range of peculiar 3-d structures formed in dispersions of hematite cubes. The formed ordering strongly depends on interactions between particles and that was tested by using coated particles and different solvents in dispersions. Also interesting re-orientation phenomena in the external magnetic field were observed caused by a permanent magnetic moment of hematite cubes.

Finally, we would like to thank Dr D. Portale and D. Detollenaere for their excellent support.

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[2] C. Graf et al., Langmuir, 19 (17), 6693 (2003)
[3] J.H.J. Thijssen, et al., Advanced Materials 18, 1662 (2006)