

MAGNETIC IRON OXIDE PARTICLES FOR COMPOSITE COATINGS

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AIM: Prepare iron oxide particles that are able to assembly within a polymeric matrix to produce surfaces with controlled hierarchical roughness.

EXPERIMENTAL:

- Colloidal magnetite particles (Fe_3O_4) prepared by co-precipitation from an aqueous solution containing $\text{Fe}^{3+}/\text{Fe}^{2+}$ ions with molar ratio of 2:1, upon addition of aqueous sodium hydroxide.
- Functionalization in aqueous dispersion at 70°C , for 1h using surfactants with various HLB values (sodium oleate, oleic acid, Spam 80 and Tween 80); product washed with ethanol and re-dispersed in isopropyl alcohol (1% solids).

CHARACTERIZATION

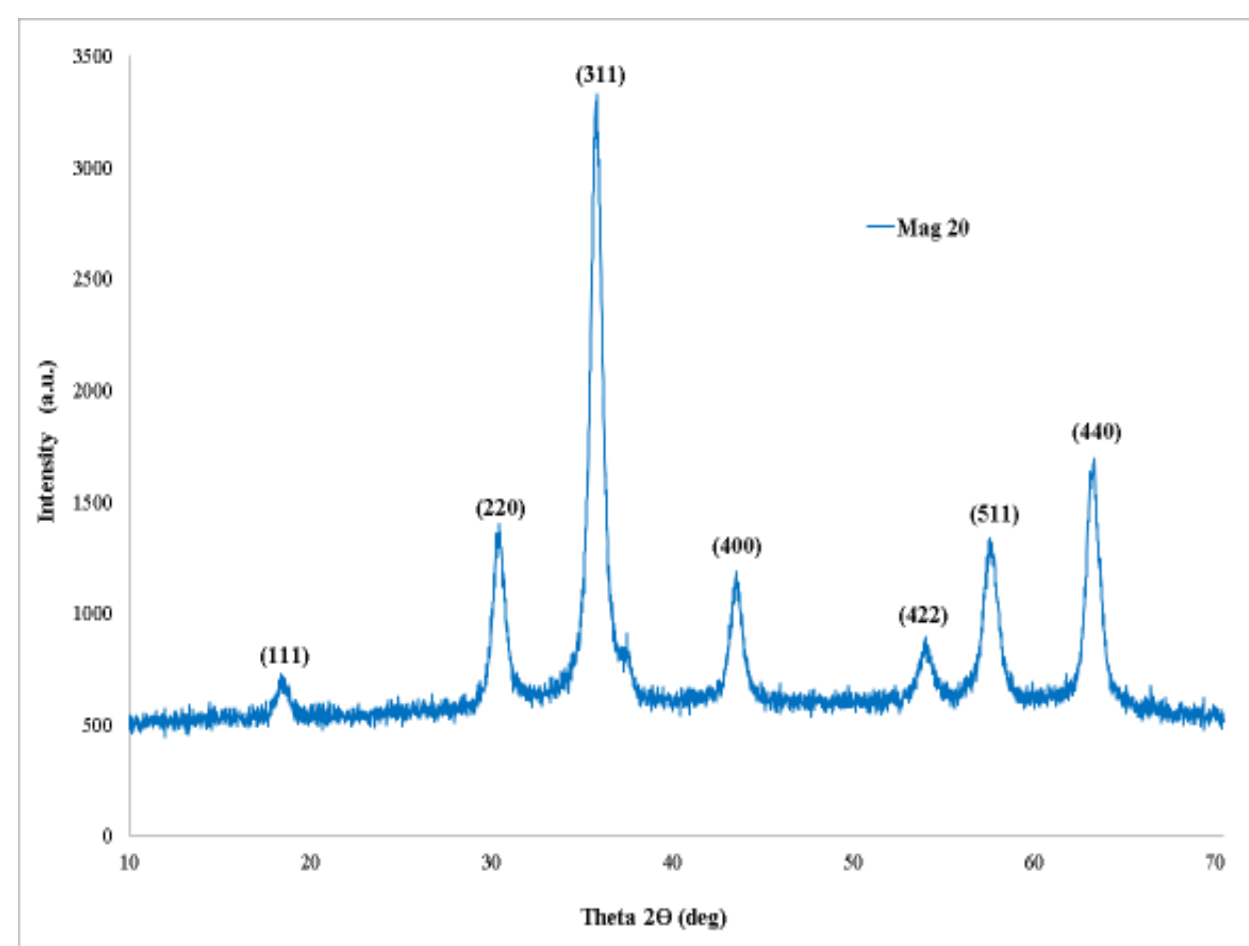


Figure 1. X-ray diffraction patterns for magnetite nanoparticles (Mag 20)

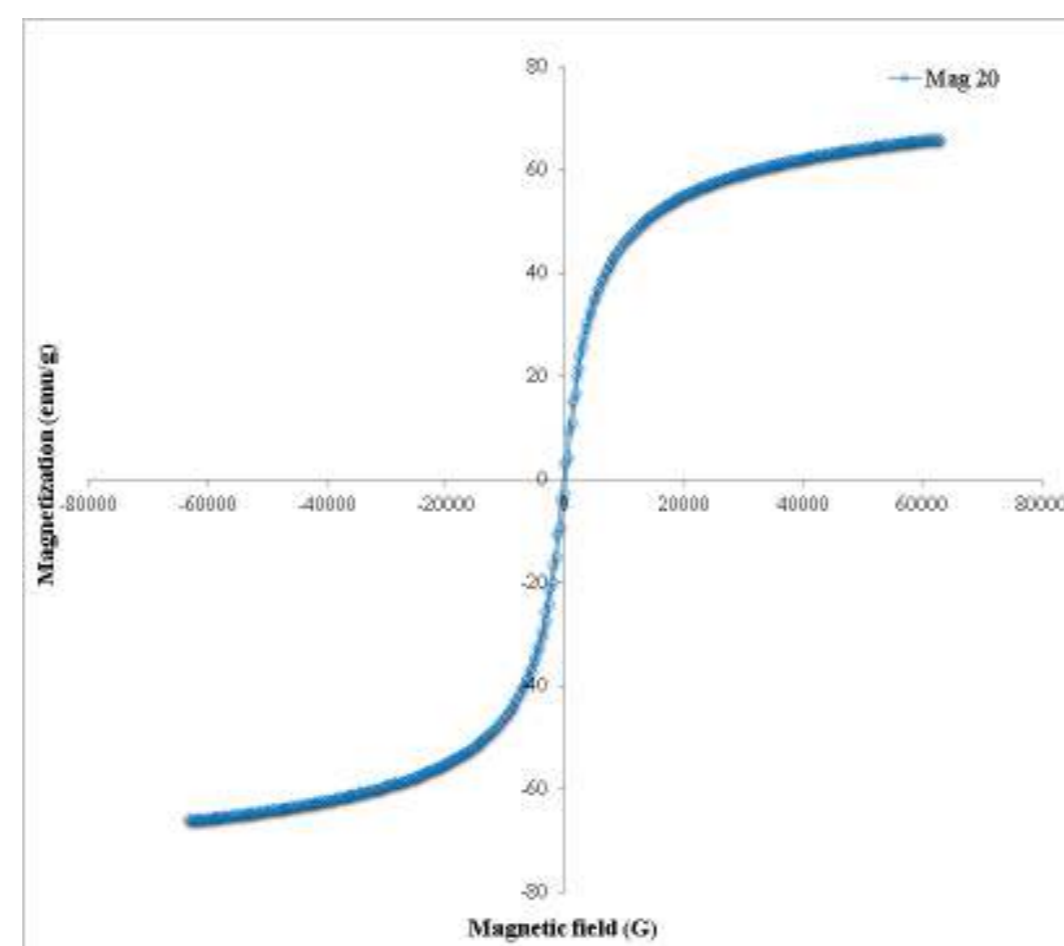


Figure 2. Magnetization measurements for magnetite nanoparticles (Mag 20)

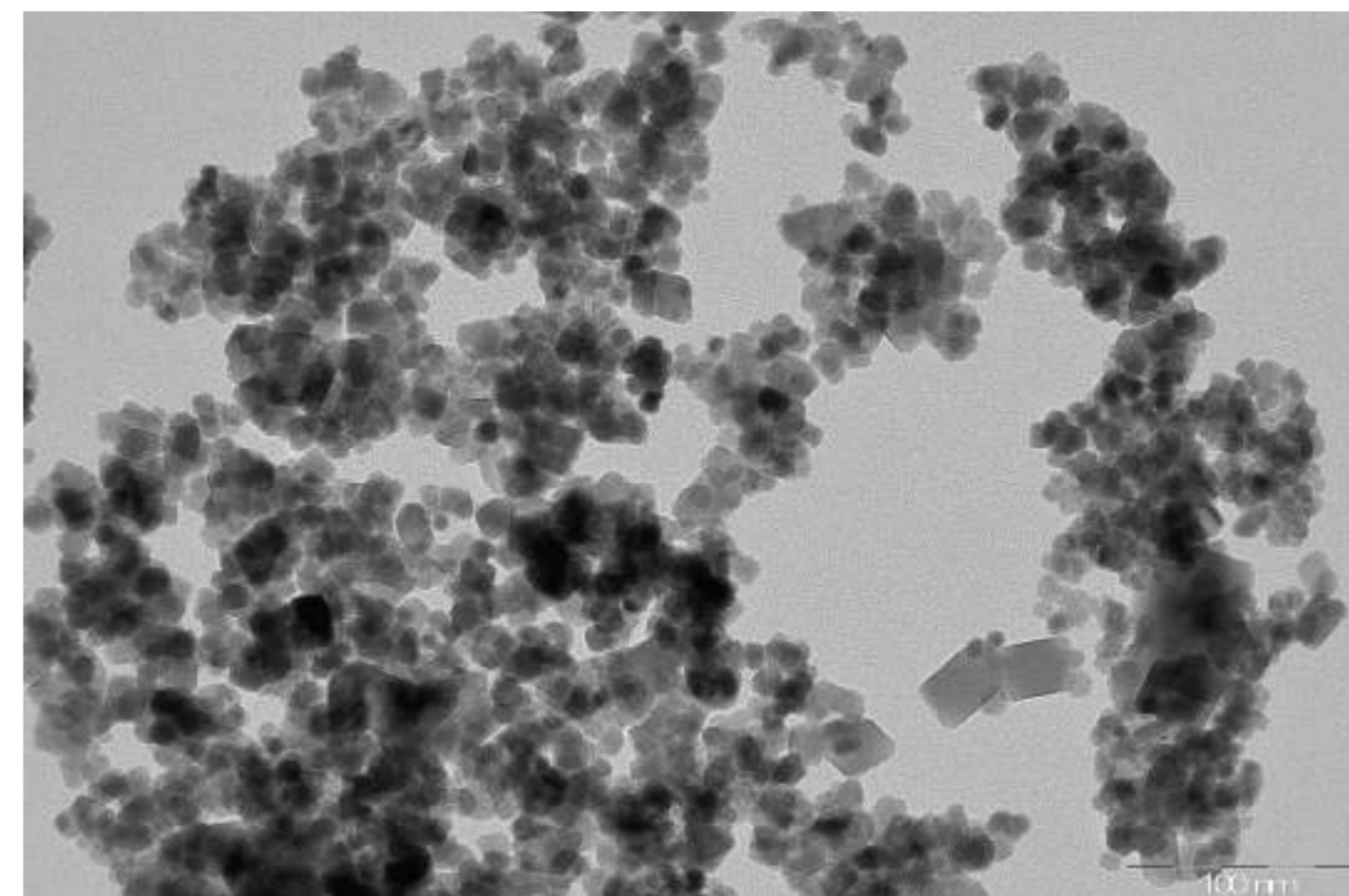


Figure 3. TEM picture of the magnetite nanoparticles (Mag 20)

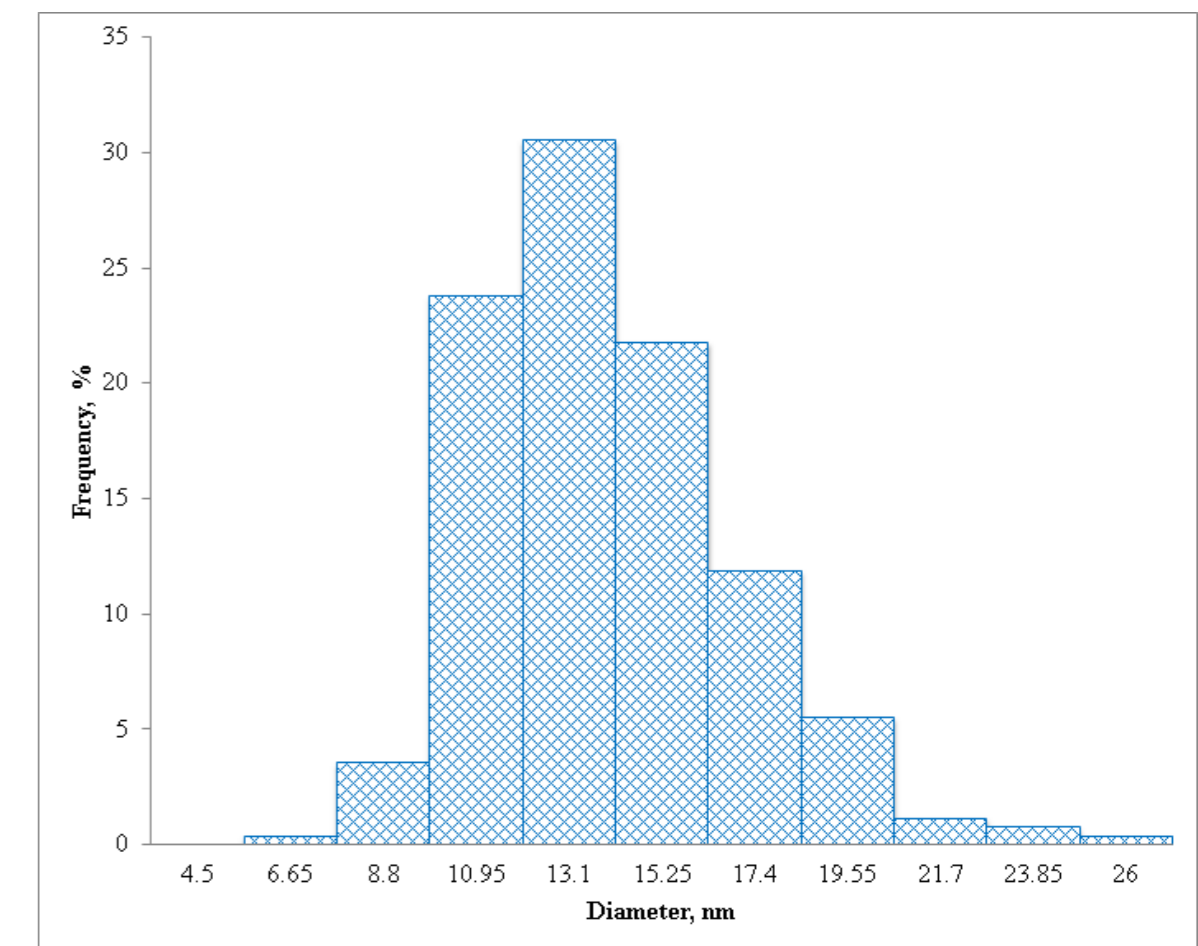


Figure 4. Size distribution of magnetite nanoparticles calculated from TEM micrographs

Batch	Magnetite suspension (1% solid), mL	Surfactant	Surfactant solution, mL	Surfactant HLB
Mag20NaOL	10	Sodium oleate, 1% (aqueous)	2	-
Mag20AcOL	10	Oleic acid, 1% (ethanol)	2	-
Mag20S80	10	Span 80, 1% (ethanol)	0,4	4,3
Mag20S/T 75/25	10	Span80, 1% 0.3ml+ Tween 80, 1% 0.1ml	0,4	7
Mag20S/T 50/50	10	Span80, 1% 0.2ml+ Tween 80, 1% 0.2ml	0,4	9,6
Mag20T80	10	Tween 80, 1% (aqueous)	0,4	15

Table 1. Functionalization study

Two-phase partition experiment

- 0.6g 1% magnetite suspension in IPA added to 10ml water and 5 ml dodecane, mixed gently and left to separate.

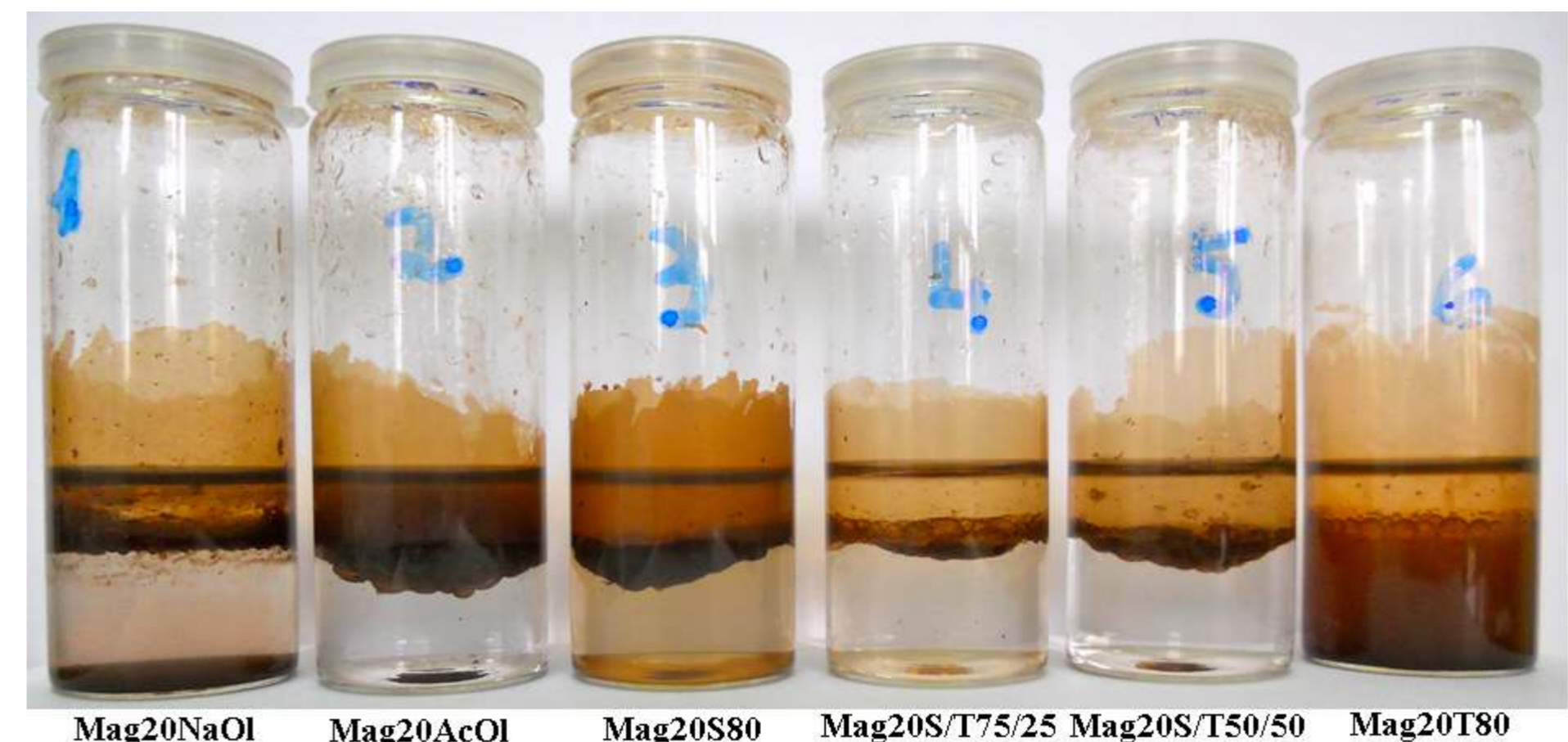


Figure 5. Two-phase partition of functionalized nanoparticles

COMPOSITE FILM PREPARATION

- 0.3g functionalized nanoparticle suspension mixed with 5ml chitosan solution (1% in acetic acid 1M), deposited by spraying on microscope glass slides); film cured by drying in magnetic field with a density of 6mT, applied perpendicular to the film surface for 20min at 75°C . Nanoparticle content in the film: approx. 6%.

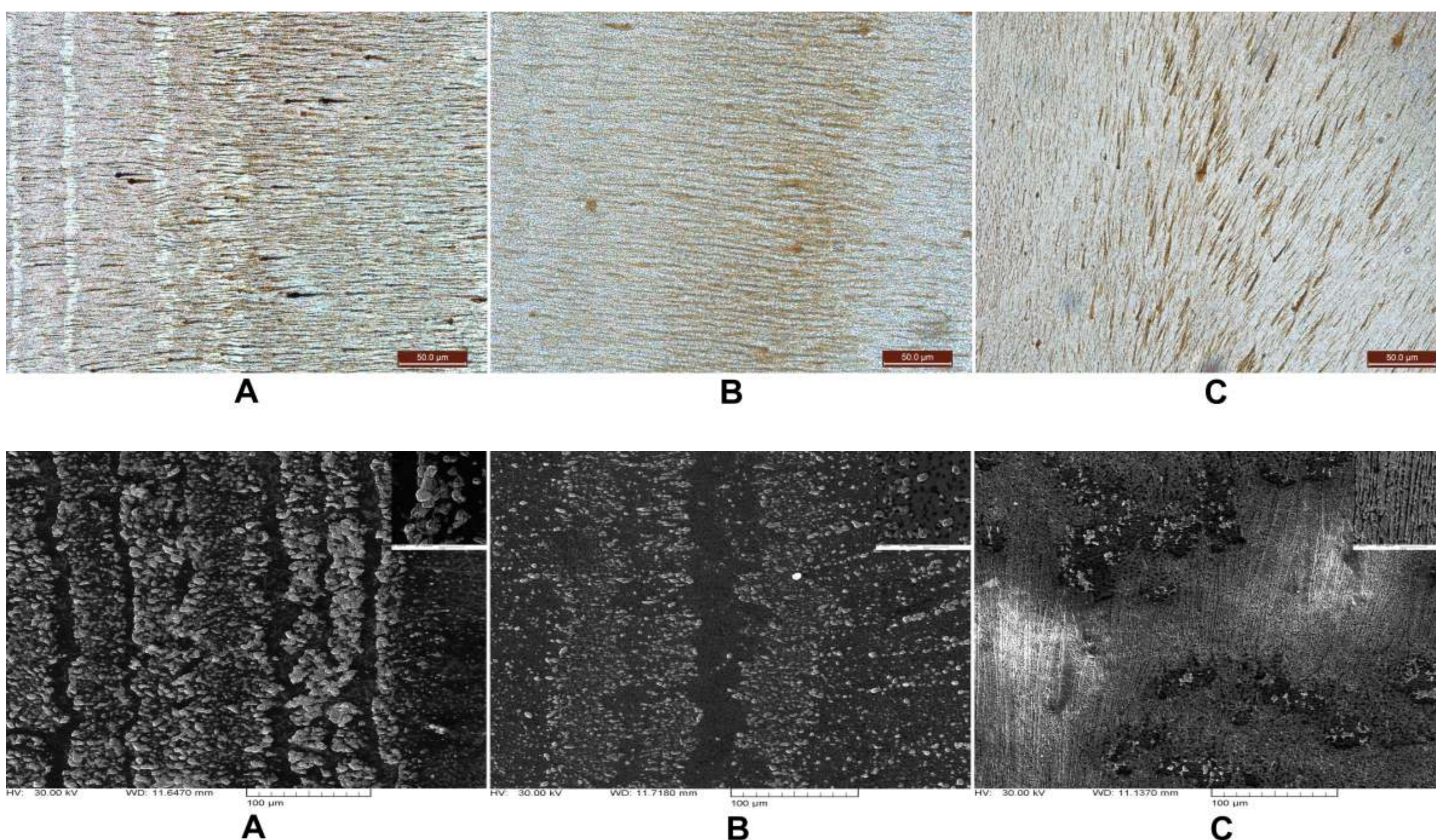


Figure 6. Optical and SEM micrographs of composite films prepared with functionalized nanoparticles.

A: Mag20AcOL; B: Mag20NaOL ; C: Mag20T80

CONCLUSIONS

- Magnetite nanoparticles with an average diameter of 13 nm and a saturation magnetization of 66 emu/g were prepared by co-precipitation in alkaline solution. They were subsequently functionalized by using surfactants with various HLB values
 - Composite functionalized nanoparticle-chitosan films prepared by spraying and cured by drying in magnetic field evidenced patterned roughness when analyzed by optical and scanning electron microscopy. The obtained texture depends on the surfactant nature.
 - The optimum film morphology was produced with the oleic acid functionalized nanoparticles, assembled in chain-like structures distributed within the polymeric film, thus creating ordered protuberances
 - Future work: modify the polymeric matrix with a low surface energy reagent to produce composite films with ice-phobic properties