Effect of Ultrasonication On Magnetic Fe_3O_4 Nanoparticles

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INTROUCTION. Iron oxide nanoparticles have gained popularity due to their easy availability, easy synthesis, and superparamagnetic properties [1]. The size of the nanoparticles ranges from 10 nm to 100 nm. Due to the inter-particle adhesion forces, nanoparticles become agglomerated, and their settlement can be observed due to the gravity forces. To achieve the maximum benefit from nanoparticles while working with them, it is desired to have an aggregate- and sediment-free structure where all the nanoparticles contribute to the dispersion. Improving the dispersion stability of nanofluids through ultrasonication has been shown to be effective. It could be noted that ultrasonication is a complicated physiochemical process, which can break down the agglomeration as well as create further aggregation, and many other effects [1]. In this work we have investigated effect of ultrasonication on magnetic Fe_3O_4 nanoparticles using different ultrasonication equipment, and effect on nanoparticles using different stabilizers and mediums

EXPERIMENT. 0,1 mg/ml Fe_3O_4 nanoparticles in water were ultrasonicated in ultrasonication bath for 15 min., collected visible sedimentation changes in tube, and measured hydrodynamic particles size by DLS. Results were compared using different ultrasonication time and ultrasonication equipment. In addition, medium was changed to PBS buffer and different concentrations of sodium acetate and trisodium citrate stabilizers were used.

NANOPARTICLES. Nanoparticles were synthesized by coprecipation method.

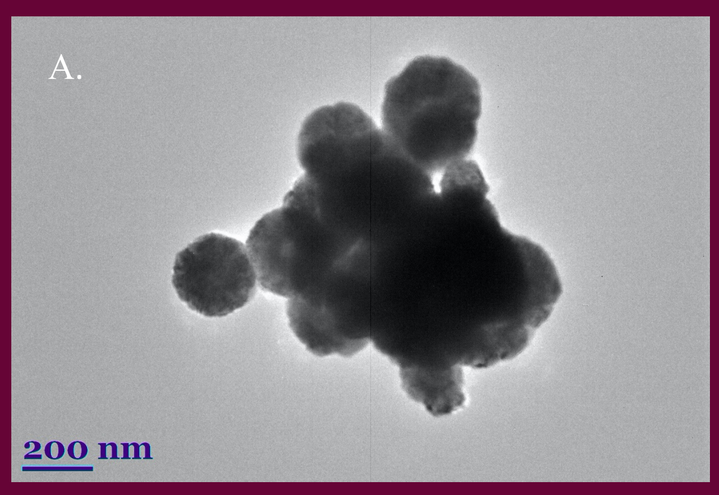

Fig. 1 Magnetic Fe_3O_4 nanoparticles synthesized by coprecipitation method ***A.*** *0,074 M $FeSO_4 \cdot 7H2O$* ***B.*** *0,074 M $FeCl_2 \cdot 4H_2O$* ***C.*** *1:2 ratio Fe^{2+}/Fe^{3+} mix*

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RESULTS.

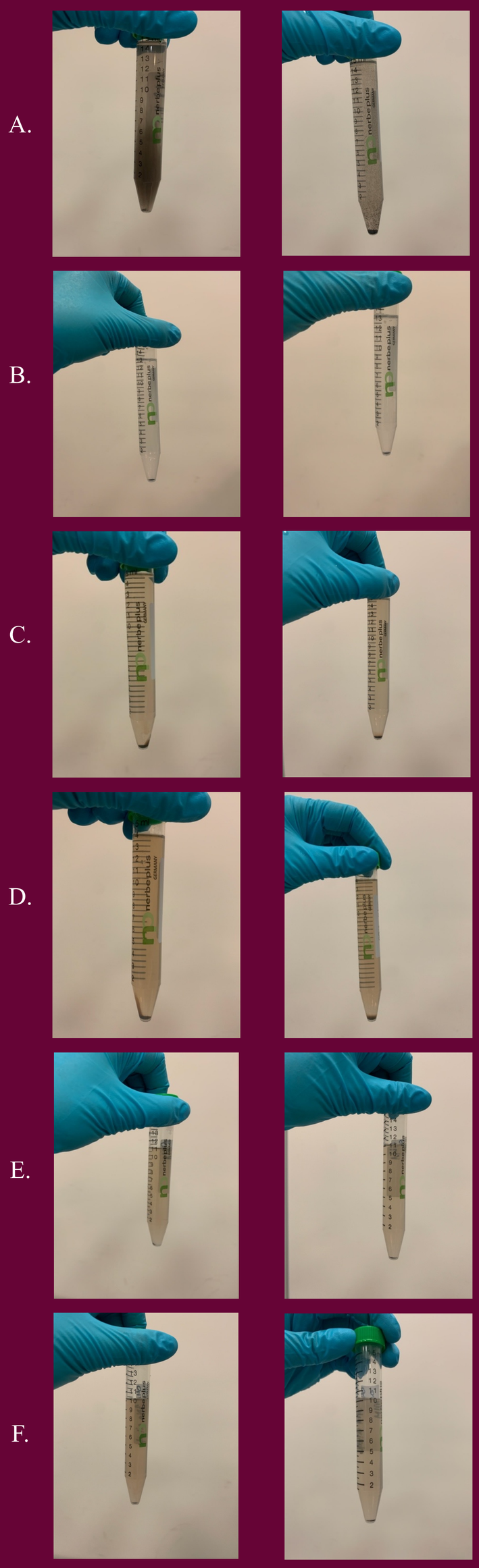

Fig. 2 Visible sedimentation change in tube after ultrasonication ***A.*** *0,074 M $FeCl_2 \cdot 4H_2O$ in H_2O* ***B.*** *0,074 M $FeCl_2 \cdot 4H_2O$ in PBS buffer* ***C.*** *0,074 M $FeSO_4 \cdot 7H_2O$ in H_2O* ***D.*** *0,024 M $FeSO_4 \cdot 7H_2O$ in H_2O* ***E.*** *$FeSO_4 \cdot 7H_2O$ with 0,01 M sodium acetate* ***F.*** *0,024 M $FeSO_4 \cdot 7H_2O$ with 0,001 M trisodium citrate*

Table 1. Size and zeta potential of the nanoparticles after ultrasonication bath from DLS data

	Size, nm	**Zeta potential, mV**
0,074 M $FeSO_4 \cdot 7H_2O$ in H_2O for **15 min**.	72,4 ± 6,44	-36,8 ± 0,74
0,074 M $FeSO_4 \cdot 7H_2O$ in H_2O for **5 min**.	98,9 ± 3,52	-54,1 ± 0,41
0,074 M $FeSO_4 \cdot 7H_2O$ in H_2O for **30 min.**	59,5 ± 3,24	-34,6 ± 0,29
0,074 M $FeSO_4 \cdot 7H_2O$ in H_2O **by ultrasonication finger**	58,0 ± 2,98	-
0,024 M $FeSO_4 \cdot 7H_2O$ **in H_2O** for 15 min.	80,4 ± 5,86	-26,2 ± 0,45
0,024 M $FeSO_4 \cdot 7H_2O$ **in PBS buffer** for 15 min.	729,8 ± 28,55	-
0,024 M $FeSO_4 \cdot 7H_2O$ with **0,01 M sodium acetate** for 15 min.	598,9 ± 66,27	-30,0 ± 0,72
0,024 M $FeSO_4 \cdot 7H_2O$ with **0,001 M trisodium citrate** for 15 min.	41,9 ± 2,76	-54,0 ± 0,41
0,074 M $FeCl_2 \cdot 4H_2O$ in H_2O for 15 min.	Too large to measure	-
0,024 M $FeCl_2 \cdot 4H_2O$ in H_2O for 15 min.	83,3 ± 2,77	-23,0 ± 0,44
1:2 ratio Fe^{2+}/Fe^{3+} mix in H_2O for 15 min.	46,3 ± 3,86	-34,5 ± 047

CONCLUSIONS.

- There is no crucial difference to use ultrasonication bath or ultrasonication finger. Visible nanoparticles sedimentation and measured size by DLS were similar.
- Different ultrasonication time shows different nanoparticles size: shorter time results bigger size nanoparticles, longer – smaller size particles.
- Nanoparticles hydrodynamic size in PBS buffer is increasing. It could be because of ions from buffer around nanoparticles.
- 0,01 M sodium acetate is not working as a stabilizer, nanoparticles are aggregated, because of it, measured size is increased. Therefore 0,001 M trisodium citrate could work as a stabilizer.
- Nanoparticles, which were synthesized using 0,074 M FeCl2·4H2O, sedimented more after 1 hour in comparison to others. In 1 hour, solution from black became transparent, big, aggregated particles were visible. It was impossible to measure particles using DLS due to their big size and fast sedimentation.
- Nanoparticles in PBS buffer were already sedimented just after ultrasonication.
- Other nanofluids after 1 hour after ultrasonication maintain solution colour unchanged and particles not sedimented.
- DLS data results are close to TEM results.