A continuous-flow process for the rapid synthesis of Fe_3O_4 nanoparticles using microwaves

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In spite of the growing demand of magnetic nanoparticles for developing applications, not much effort has been devoted to transfer laboratory knowledge into large-scale production schemes. Continuous processes are always preferred over batch ones when reproducible and scalable industrial procedures are needed. For instance, the production of magnetite nanoparticles by oxidative precipitation of $FeSO_4$ in aqueous media has been demonstrated by a continuous approach that offers (i) the complete separation of the green rust's precipitation from Fe_3O_4 nucleation, (ii) constant concentrations in all ionic and solid forms, and (iii) the possibility to control critical parameters, through on-line regulation of synthesis parameters such as the reactor's pH. However, this process requires a long ageing period of at least 4 h during which reaction mixture remains in a heated bath (90 ^oC). This step appears to have significant impact in the total energy consumption while the high residence time implies to the need for larger facilities. To overcome such issues, a different setup which introduces a microwave heating step was designed. Here, the ageing reactor (CSTR) was placed into a microwave oven and continuously fed with the green rust precursor. Following this scheme, Fe_3O_4 nanoparticles with diameter around 30 nm were successfully produced by applying a residence period of less than 10 min in a polyacetal tank. Surprisingly, by using a plug-flow reactor (PFR), a very high heating rate was succeeded and well-defined magnetic nanoparticles were received with a residence period of less than 30 s. The suggested setup provides an automatable solution for onsite nanoparticle synthesis in the biomedical sector by non-specialized staff.

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