Particuology 26 (2016) 114–117

Contents lists available at ScienceDirect

Particuology

journal homepage: www.elsevier.com/locate/partic

Short communication

Rapid determination of iron oxide content in magnetically modified particulate materials

Ivo Safarik[a,b,c,*], Leona Nydlova[a], Kristyna Pospiskova[b], Eva Baldikova[a,d], Zdenka Maderova[a], Mirka Safarikova[a,c]

[a] Department of Nanobiotechnology, Institute of Nanobiology and Structural Biology of GCRC, Na Sadkach 7, 370 05 Ceske Budejovice, Czech Republic
[b] Regional Centre of Advanced Technologies and Materials, Palacky University, Slechtitelu 27, 783 71 Olomouc, Czech Republic
[c] Department of Nanobiotechnology, Biology Centre, CAS, Na Sadkach 7, 370 05 Ceske Budejovice, Czech Republic
[d] Department of Applied Chemistry, Faculty of Agriculture, University of South Bohemia, Branisovska 1457, 370 05 Ceske Budejovice, Czech Republic

ARTICLE INFO

Article history:
Received 6 October 2015
Received in revised form 9 November 2015
Accepted 18 November 2015
Available online 18 January 2016

Keywords:
Magnetic permeability meter
Magnetic iron oxides
Magnetically modified materials

ABSTRACT

Magnetically responsive composite materials have been used in interesting applications in various areas of bioscience, biotechnology, and environmental technology. In this work, a simple method to determine the amount of magnetic iron oxide nano- and microparticles attached to magnetically-modified particulate diamagnetic materials has been developed using a commercially available magnetic permeability meter. The procedure is fast and enables dry particulate magnetically modified materials to be analysed without any modification or pretreatment. We show that the magnetic permeability can be measured for materials containing up to 20% magnetic iron oxide. The magnetic permeability measurements are highly reproducible.

Introduction

Magnetically responsive nano- and microparticles have been used in many important applications in various areas of bioscience, medicine, biotechnology, and environmental technology. Such materials exhibit several types of responses to an external magnetic field, such as selective separation, targeting and localization, heat generation in high frequency alternating magnetic fields, increases in negative T2 contrast by magnetic iron oxides nanoparticles during magnetic resonance imaging, or great increases in apparent viscosity of magnetorheological fluids when subjected to a magnetic field (Safarik, Pospiskova, Horska, Maderova, & Safarikova, 2014). Currently, large amounts of diverse magnetic nano- and micromaterials can be obtained commercially, or can be produced in research laboratories using many different basic principles (Laurent et al., 2008; Kharissova, Dias, & Kharisov, 2015).

In the last two decades, a large number of magnetically responsive materials, prepared from a huge variety of diamagnetic ("nonmagnetic") particulate materials with interesting properties, have been described. Various modification techniques can be employed to convert nonmagnetic materials into magnetic derivatives (Safarik, Horska, Pospiskova, & Safarikova, 2012a). Recently, two extremely simple and widely applicable magnetic modification procedures have been developed based on the interaction of microwave synthesized magnetic iron oxide nano- and microparticles or perchloric acid stabilized magnetic fluid with modified diamagnetic materials. After thorough drying, stable magnetically responsive composite materials are formed. The magnetic iron oxide particles are mainly localized on the surface or within the pores of the treated materials, and their mutual interaction is quite strong and stable. The strong binding of magnetic iron oxide particles to nonmagnetic materials is thought to arise from a subtle balance of van der Waals, electrostatic, and hydrophobic interactions between the magnetic particles and the treated material (Saito, Koopal, Nagasaki, & Tanaka, 2008). The magnetic properties of the composites can be tuned simply by changing the amount of magnetic modifier used (Safarik & Safarikova, 2014; Safarik, Horska, Pospiskova, & Safarikova, 2012b). The prepared magnetic composites can be magnetically separated very easily using either NdFeB permanent magnets or commercially available magnetic separators (see Fig. 1).

Large amounts of magnetically responsive materials with different degrees of magnetic modification have been prepared in our lab in quantities ranging from grams to approximately one

* Corresponding author at: Prof. Ivo Safarik, Biology Centre, Czech Academy of Sciences, Department of Nanobiotechnology, Na Sadkach 7, 370 05 Ceske Budejovice, Czech Republic. Tel.: +420 387775608; fax: +420 385310133.
E-mail address: ivosaf@yahoo.com (I. Safarik).

http://dx.doi.org/10.1016/j.partic.2015.11.001
1674-2001/

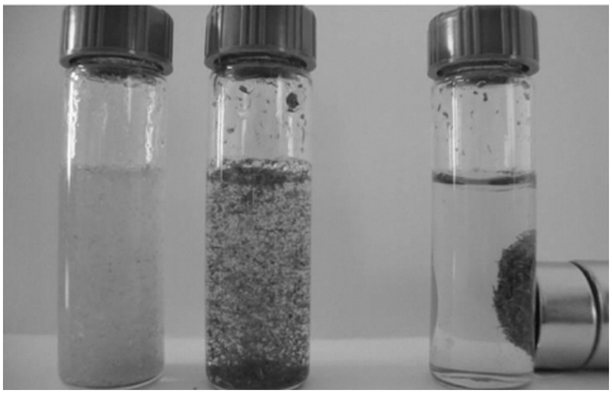

Fig. 1. Appearance of original straw particle suspension (left), suspension of straw after magnetic modification with microwave synthesized magnetic iron oxide particles (middle), and demonstration of magnetic separation of magnetically modified straw (right).

kilogram using both procedures. It became obvious that a rapid method enabling the measurement of the magnetic particle content of the modified materials was needed to confirm the proper magnetic modification of the final materials and to determine iron oxide content in mixtures prepared from materials with different levels of magnetic label coating. Thus, in this paper, a simple procedure to measure the amount of magnetic iron oxide on magnetically modified materials has been developed using a commercially available magnetic permeability meter. The method is rapid and allows dry powdered magnetically modified materials to be used for measurements without any modification.

Experimental

Materials

Bentonite clay was obtained from Tamda (Olomouc, Czech Republic), and montmorillonite, powdered magnetite, ferrous sulphate heptahydrate, and sodium hydroxide were obtained from Sigma-Aldrich (St. Louis, MO, USA). Oak sawdust and milled rye straw (about 0.1–2 mm in diameter) as well as potato starch were obtained locally. Water-based magnetic fluid stabilized with perchloric acid was prepared using a standard procedure (Massart, 1981). The ferrofluid was composed of magnetic iron oxide nanoparticles with diameters ranging between 10 and 20 nm (electron microscopy measurements). The relative magnetic fluid concentration (30.2 mg/mL) is given as the iron (II, III) oxide content determined by a colorimetric method (Kiwada, Sato, Yamada, & Kato, 1986).

Magnetic modification with microwave synthesized magnetic iron oxide particles

Nonmagnetic materials were magnetically modified using microwave-synthesized magnetic iron oxide particles (Safarik & Safarikova, 2014). In a typical procedure, 1 g $FeSO_4{\cdot}7H_2O$ was dissolved in 100 mL of water in a 600–800 mL beaker and sodium hydroxide solution (1 mol/L) was added slowly under mixing until the pH reached ca. 12; during this process a precipitate of iron hydroxides was formed. Then, the suspension was diluted to 200 mL with water and inserted into a standard kitchen microwave oven (700 W, 2450 MHz). The suspension was usually treated for 10 min at the maximum power of the oven. The beaker was then removed from the oven and the formed magnetic iron oxide nano- and microparticles were repeatedly washed with water until neutral pH was reached.

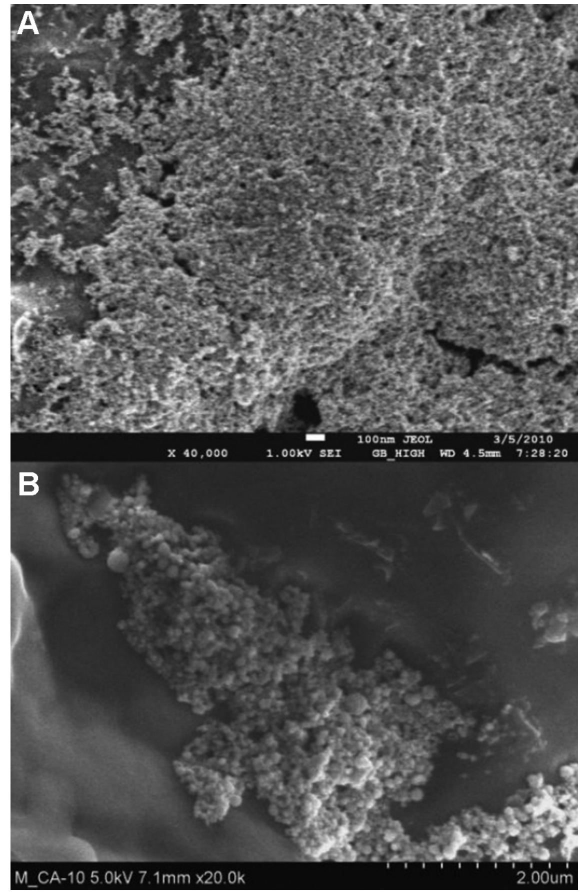

Fig. 2. Scanning electron microscope images of lignocellulosic materials modified with magnetic fluid (a) and with microwave-synthesized magnetic iron oxide nanoparticles (b).

To prepare magnetically responsive materials, 1 g of the target powdered material was thoroughly mixed in a short test-tube or a small beaker with an appropriate amount of microwave iron oxide nano- and microparticle suspension (one part completely sedimented iron oxide particles and four parts water; the exact iron oxide content was determined after drying to constant weight). Vigorous mixing with a spatula or laboratory spoon enabled homogeneous distribution of the magnetic nanoparticles and microparticles within the treated material. The mixture was allowed to dry completely at temperatures usually not exceeding 60 °C for 48 h. To change the magnetic response of the modified material, the amount of iron oxide particles (i.e., the volume of iron oxide suspension) was changed as required (Safarik & Safarikova, 2014). For specific materials (e.g., bentonite), the iron oxide particle suspension was prepared in ethanol.

Magnetic modification with magnetic fluid

In a typical procedure, 1 g of the powder to be modified was thoroughly mixed in a short test-tube or a small beaker with an appropriate amount (e.g., 1 mL) of water based ferrofluid stabilized with perchloric acid. Mixing with a spatula or laboratory spoon enabled homogeneous distribution of the magnetic fluid within the treated material. The mixture was allowed to dry completely at temperatures usually not exceeding 50–60 °C for 48 h (Safarik et al., 2012b). Use of a larger volume of magnetic fluid led to the formation of modified materials with higher magnetic responses.