PREPARATION OF MAGNETIC NANO PARTICLES ON CELLULOSE FIBRES

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Effects of the electromagnetic radiation have been a concern for some years now, especially due to the growing presence of mobile telephones and other electronic devices, used in everyday life. There are numerous shielding materials available to protect from electrosmog, mostly all of them consisting of metal, metal alloys or having metal components. Aim of our research is to develop a shielding material based on cellulose fibres, with magnetic iron particles as the active component for absorption of electromagnetic radiation.

Iron magnetic particles were precipitated from the solutions of iron salts in the presence of ammonia as a catalyst. Surface of resultant fibers with layers of nano particles was characterized with a scanning electron microscope. Mechanical properties were also checked.

Iron particles-covered cellulose fibers can represent a versatile shielding material, since they could be processed and formed into different forms of covers and shields, depending on the end application.

1 INTRODUCTION

It is not possible to imagine modern society without the electricity as it plays a major role in our everyday life. Every time we use electricity and electrical appliances, we are exposed to electric and magnetic fields, which are basically forces surrounding electrical equipment, power cords and wires, carrying electricity, including outdoor power lines. Electric field is created whenever a wire is plugged into an outlet, regardless of whether the appliance is turned on or not. Increasing of voltage results in a stronger electric field. Magnetic fields are created when an electric current is flowing within a device or a wire. The greater the current, the stronger the magnetic field.

Although no direct and definitive link between exposure to electromagnetic fields (EMF) and occurrence of serious illnesses (such as childhood cancer; investigation of which and its connection to electromagnetic field in 1979 by Wertheimer and Leeper brought this subject to prominence [1]) has been established, there are reports describing various consequences of sensitivity to EMF exposure. Health problems range from headaches, tiredness, behaviour and mod changes, nose bleeds, depression and concentration problems. A certain degree of protection from electromagnetic smog is clearly needed, especially with the rapidly increasing number of cellular phones, computers connecting to the internet wirelessly, WiFi stations, with which we are encountered daily.

Shielding from/of electromagnetic radiation is necessary in other areas, as well; military being one of the examples, where a proper shielding material would conceal electromagnetic waves, emitted by military equipment and on the other hand, protect equipment from EMF exposure, preventing electromagnetic interference.

Aim of our study is to develop composite shielding materials with regenerated cellulose fibres as the base material and magnetic nano particles as the active component. Magnetic nano particles, specifically magnetite (Fe_3O_4), were precipitated in the presence of viscose fibres, thus forming a magnetic layer on the fibres' surface, rendering them magnetic. Such a composite fibre is expected to absorb electromagnetic waves.

In the presentation we have compared the morphology of magnetic layers formed on fibres via different synthesis routes as well as determined the mechanical properties of the composite fibres in order to determine the effect of the synthesis conditions on the fibres' overall performance.

2 EXPERIMENTAL

Magnetite is one of the most widely used magnetic pigments in the production of magnetic recording and information storage media. It is a ferrimagnetic material with a IUPAC name *iron (II,III) oxide*. It's chemical formula Fe_3O_4 can also be written as $FeO.Fe_2O_3$, which is one part *wüstite* (FeO) and one part *hematite* (Fe_2O_3).

As a starting material, regular viscose fibres produced by Lenzing AG, were used.

Two procedures were used for synthesis of magnetite (Fe_3O_4) particles and for magnetite-covered regenerated cellulose fibres; one according to Massart [2] and a modified Massart's procedure by Claesson [3].

Procedure A ([2]): aqueous solution of ferric chloride ($FeCl_3$) and solution of ferrous chloride ($FeCl_2$), dissolved in HCl, is added to ammonia solution during stirring. Magnetic particles are isolated from the solution by magnetic decantation. The method is based on the stoichiometric mixture of Fe^{2+} and Fe^{3+} in aqueous media and the coprecipitation of the corresponding hydroxides [$Fe(OH)_2$ and $Fe(OH)_3$] upon the addition of a strong alkali [4]. Under vigorous stirring relatively fast aging of those hydroxides results in the formation of magnetite. Viota et al. [5] determined the average size of the particles, formed via Massart's procedure, to be 8±3 nm.

Procedure B ([3]): this procedure differs from the one of Massart by replacing the aqueous solution of hydrochloric acid as a solvent for ferrous chloride ($FeCl_2$) with water. Iron chloride salts ($FeCl_3$ and $FeCl_2$) are dissolved in water and under vigorous stirring, ammonia solution is added. In our case we reversed the addition of components of a reaction mixture; solution of iron chloride salts was added to the ammonia solution.

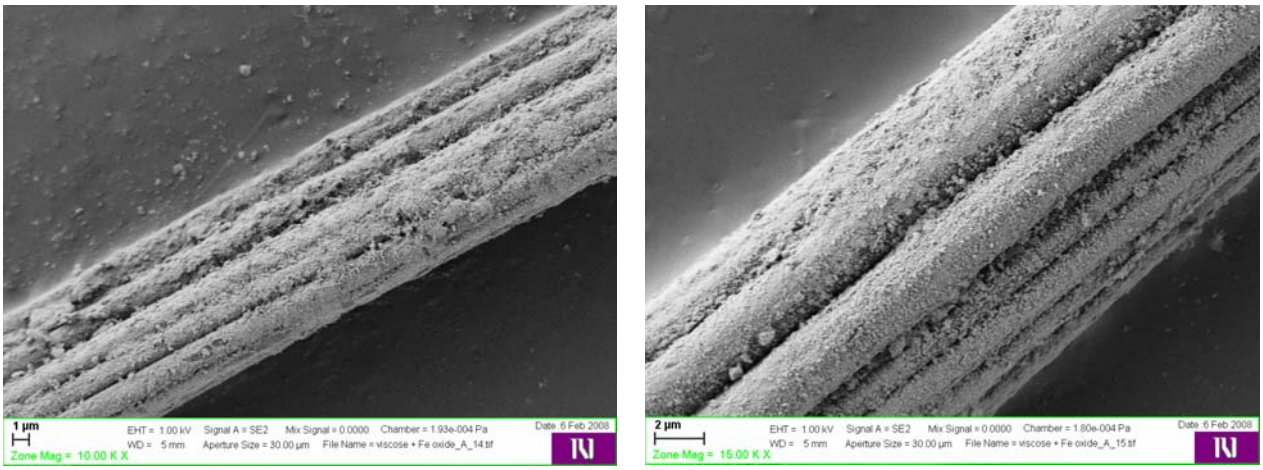
In order to precipitate magnetite particles on the fibres' surface, we introduced regenerated cellulose fibres into the reaction mixture in both procedures before the formation of Fe_3O_4 took place. Fibres were immersed in the aqueous solution of ammonia and stirred for some time, ensuring that the ammonia, with its swelling ability for cellulose substrates, penetrated into fibres' inner structure. It was only then that the two solutions of iron chloride salts were added to the mixture. After twenty minutes of stirring we put the vessel with fibres and formed particles aside and placed it onto a magnet. Magnetite-covered fibres and magnetite particles sedimented due to the attraction of the magnet and surplus solution was removed. Fe_3O_4-covered fibres were thoroughly rinsed with distilled water and dried in an oven at 60°C.

Magnetite-covered viscose fibres, prepared via procedure A, were designated as *viscose+Fe oxide_A* and fibres with a magnetite layer, prepared according to procedure B, as *viscose+Fe oxide_B*.

3 RESULTS

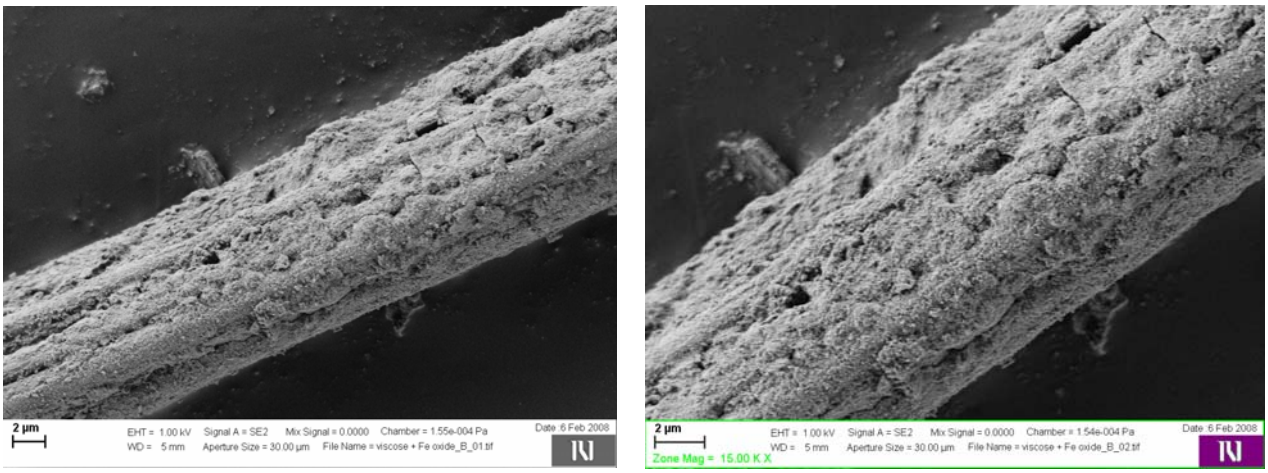
3.1 SEM images of magnetite-covered viscose fibres

Electron microscope FE-SEM SUPRA 35 VP (Carl Zeiss) was used for observation of surface morphologies of fibre samples. Figure 1 shows surfaces of viscose fibres covered with a magnetite layer according to procedure A. Figure 2 shows surfaces of viscose fibres covered with a magnetite layer according to procedure B.



a b

Figure 1: a) sample *viscose+Fe oxide_A* at 10 000 X magnification b) sample *viscose+Fe oxide_A* at 15 000 X magnification



a b

Figure 2: a) sample *viscose+Fe oxide_B* at 10 000 X magnification b) sample *viscose+Fe oxide_B* at 15 000 X magnification

Magnetite particles in both cases precipitate in quite thick, uneven layers. While it is reported [5], as was mentioned above, that the average size of the magnetite particles, synthesized through Massart's procedure, is 8±3 nm, this value represents the dimensions of particles in the ferrofluid, that is, dispersed and stabilized in an appropriate medium. When dried, Fe_3O_4 particles tend to aggregate and form larger clusters, as it can be seen on the surfaces of covered fibres, resulting in much larger dimensions. While sample *viscose+Fe oxide_A* shows a more homogenous coverage and distribution of particles on its surface, that is not the case with a sample *viscose+Fe oxide_B*. Thick clusters of agglomerated particles cover the fibres surface, forming an uneven crust (Fig. 2). It is not likely that this result is due to the slightly different synthesis procedure (water as solvent as opposed to HCl) but more likely to the more pronounced effect of the capillary force between particles. Magnetite-covered viscose fibres were left to sediment on magnet after the synthesis

was over along with surplus formed particles in the solution; those particles were deposited on the already formed layers of magnetite on the fibres' surface during sedimentation and after the liquid media between them was removed during drying, they strongly attached to the magnetite-covered fibres, thus resulting in large clusters.

3.2 Mechanical properties of magnetite-covered viscose fibres

In order to determine the effect of the synthesis conditions during the formation of particles on the fibres' mechanical properties, especially from the point of view of acid addition (procedure A), tenacity, elongation and force at break were determined.

A Vibroskop 400/Vibrodyn 400 (Lenzing) apparatus for testing of tensile properties of fibres was used for determination of some basic mechanical properties of untreated and magnetite-covered viscose fibres (Table 1). Testing of fibres' mechanical properties was carried out under standard climatic conditions; 25±2°C and 65±2% of relative humidity.

samples	titer [dtex]	tenacity [cN/tex]	elongation [%]	breaking force [cN]
untreted	1,41	22,3	19,6	3,15
viscose+Fe oxide_A	1,56	21,3	18,6	3,31
viscose+Fe oxide_B	1,50	21,5	19,6	3,23

Results show that the synthesis of magnetite particles in the presence of viscose fibres does not significantly deteriorate the mechanical properties of used cellulose substrate and its overall performance. There is only a slight decrease of the fibres' tenacity and in the case of sample *viscose+Fe oxide_A* a decrease in value of elongation at break. Magnetite coatings increase the modified fibres fineness and their breaking force.

4 CONCLUSION

It can be concluded from the presented preliminary results that the procedure used for the preparation of magnetite-covered viscose fibres yields a magnetic cellulose fibres which is attracted to the permanent magnet. Synthesis conditions do not decrease the fibres mechanical performance, since despite the addition of an acid solution of an iron chloride salt, reaction still proceeds in the alkaline medium, due to the excess of the ammonia solution. Precipitated layers of magnetite particles are uneven and inhomogeneous, due to the large clusters formed on the fibres surface. One of the reasons for this to happen is probably a rapid formation of magnetite particles. Optimising of the synthesis conditions to achieve more homogenous layers is one of the next steps in our research and after that, extensive characterisation of composite fibres' magnetic properties and ability to absorb electromagnetic radiation will be performed.

5 REFERENCES

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