O8-2	**Applications of encapsulated actives**	

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INTRODUCTION

During the last decades more and more attention has been paid to the environmental effects of the many different actives added to a wide variety of products. This includes e.g. addition of biocides to outdoor wood paint and coating as well as to plastics and textiles for outdoor use in order to prevent growth of fungi and algae; addition of flame retardants to plastics for electronics and textiles for indoor use; addition of dyes and UV stabilizers to prolong aesthetics of products, etc. These additives may release from the products to the surrounding environment which has resulted in more and stricter legislation on the use of many additives. To follow this movement the industry has to develop new and environmentally friendly ways to protect their products. The coatings industry is a part of this movement along with other industries such as textile, plastics, and building materials. A significant problem in using additives is the undesired high release of additive to the environment. By changing the traditional way of adding e.g. biocides, flame retardants and stabilizers to materials this high release of chemicals can be minimized.

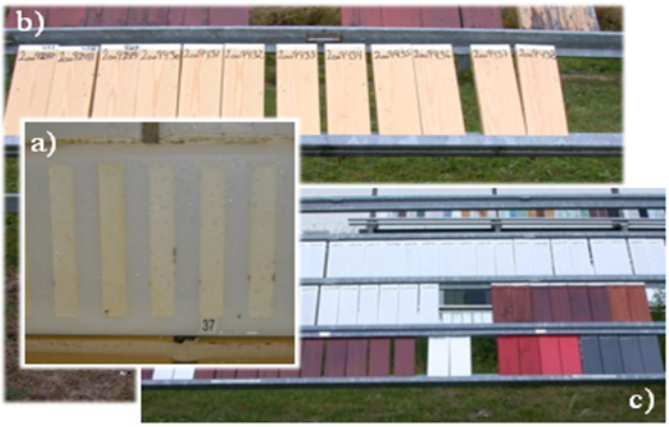

Figure 1 : Tests of different applications for encapsulated actives. a) is indoor humid panel tests of caulking compounds, while b) and c) are outdoor exposures of wood paints and coatings.

To follow this change towards greener products a solution can be the use of encapsulation technologies. By encapsulating additives the active will be released in a controlled manner depending on the encapsulation method chosen. This choice relies on both the actual additive, the purpose of the additive as well as the matrix it is added to. This will enable a specifically designed capsule to release the additive at desired rate or desired time when needed. Such carefully designed capsules will minimize the use of unwanted chemicals, at the same time maximizing the effect of the additive, and eventually minimize environmental effects. With respects to the examples given, less biocide will be washed off the coatings, plastics and textiles, and flame retardants will not release from plastics or textile until the case of a fire. Also, prolonged life times of plastic and textile products will be obtained when dyes or stabilizers slowly release from a protected environment in the capsules. The latter adds yet another aspect to the importance of using encapsulated additives since longer life times reduce the use of petrochemically derived products and thereby reduces CO_2 emissions from production, and dependence on petroleum supply.

In this study silica particles were incorporated into three matrices to provide a controlled release of actives. Encapsulated biocides were added to paint and caulking compounds to reduce the loss of biocide in wet environments as well as provide UV protection, and an encapsulated UV stabilizer was added to poly(propylene) to control the release of the stabilizer. In all three cases a more or less pronounced effect of encapsulation was found, resulting in improved UV stability and controlled release in accelerated tests.

MATERIALS AND METHODS

Preparation of particles

Silica particles with 3-iodoprop-2-ynyl *N*-butylcarbamate (IPBC) have been produced using sol-gel synthesis (Sørensen 2010, patent application 09150746.7-2103), the microparticle procedure a slightly modified version of procedures described in the literature (Sun 2003, Botterhuis 2006), and modified nanoparticles (Huo 2006). Particles containing other actives described here, namely carbendazim, 2-*n*-octyl-4-isothiazolin-3-one (OIT) and Tinuvin P, have been synthesized using same procedure, substituting IPBC with the active.

Characterization

The average size of microparticles and aggregates as well as the size of polydispersity was determined by laser diffraction, using a Malvern Mastersizer 2000 (Malvern Instruments Ltd., Malvern, UK). The instrument was calibrated using standard latex microparticles.

Selected microparticles have been characterized by scanning electron microscopy (SEM) on a JEOL scanning electron microscope (JEOL, Tokyo, Japan) or a Zeiss Gemini Ultra 55 (Carl Zeiss, Oberkochen, Germany). For determination of the inner structure of the particles Fast Ion Bombardment (FIB) was applied in combination with

SEM, i.e. using FIB-SEM Zeiss 1540EsB crossbeam (Carl Zeiss, Oberkochen, Germany).

Nanoparticles have been characterized using dynamic light scattering (DLS) giving particle size distributions using a Malvern Zetasizer Nano (Malvern Instruments Ltd., Malvern, UK).

RESULTS AND DISCUSSION

Spherical silica particles containing different actives have been synthesized. Particle sizes range from nanoparticles with diameters of ca. 20-25 nm as seen by DLS measurements (Fig. 2) to microparticles of 1-5 µm diameters.

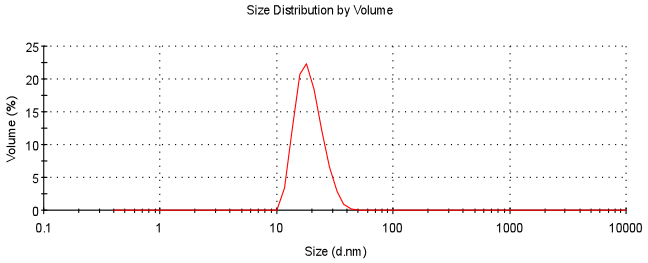

Figure 2 : DLS measurement of nanoparticles containing IPBC.

The sizes of spherical microparticles have been estimated from SEM and FIB-SEM (Fig. 3). The porous particles contain 25-35 weight % biocides, e.g. IPBC, OIT or carbendazim. They can be suspendede well in e.g. wood paints and caulking compounds using mixing methods as usually applied for such products. Likewise, silica particles containing 31 % of the UV stabilizer Tinuvin P have been produced and incorporated into poly(propylene).

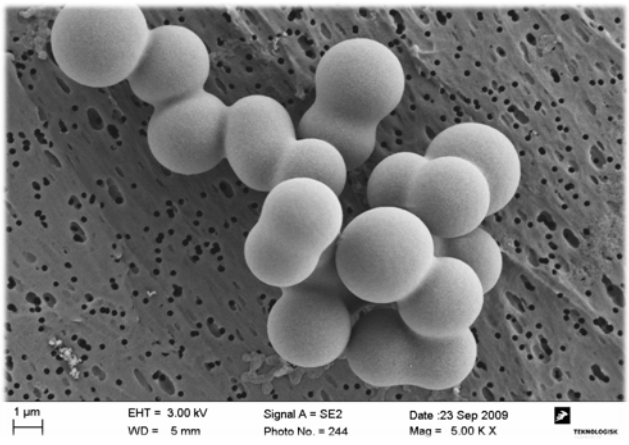

Figure 3 : SEM micrograph of spherical porous microparticles containing Tinuvin P.

Preliminary studies with both biocides and UV stabilizer show UV protection as well as significantly delayed and prolonged release of the actives. In Fig. 4 data on an accelerated release study of the UV stabilizer Tinuvin P is given. It is seen that encapsulated Tinuvin P is significantly slower released from poly(propylene) than free Tinuvin P. This preliminary study has been performed with several solvents, all showing tendencies for encapsulated Tinuvin P releasing slower than free Tinuvin P.

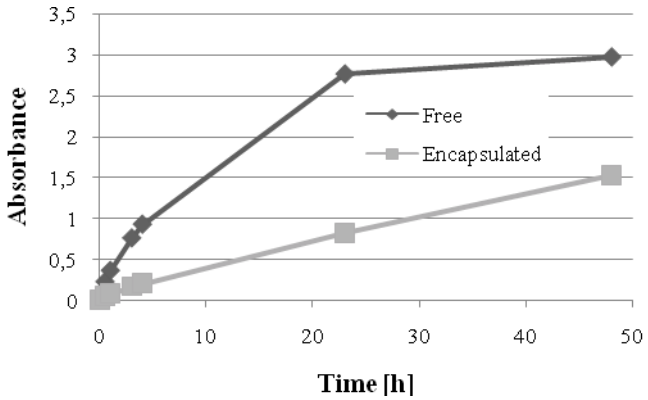

Figure 4 : Graph illustrating delayed release of encapsulated Tinuvin P compared to free Tinuvin P from (propylene) to toluene.

Biological evaluations of the performance of paints and coatings with encapsulated IPBC in accelerated ageing tests show the possibility of producing carefully designed particles that both perform slow release and protect against UV degradation of the biocide.

CONCLUSIONS

Studies of encapsulated biocides show the possibility of designing particles suited for slow release of biocide that will prolong life time of coatings or make it possible to produce well-performing coatings that contain less biocide and are thereby more environmentally friendly.

Likewise, panel tests of caulking compounds with encapsulated biocides show promising results, and preliminary testing of UV stabilizers in poly(propylene) demonstrates significantly delayed release.

Further studies are necessary and ongoing to obtain deeper knowledge of the encapsulated actives and their performance in different matrices.

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