TMA – elucidating the film formation of the dry coating process

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The dry coating process is an alternative to the conventional coating technology avoiding completely the use of organic solvent and water. The process can be divided into two phases: the coating and the curing step. During the coating step polymer powder and liquid plasticizer are added separately but simultaneously to the pellets and adhere on their surface. During the curing phase film formation occurs by increasing the temperature (1).

Conducting the dry coating process no water with plasticizing quality has to be evaporated because no volatile plasticizer is in use. Though, due to the absence of water no temporarily plasticizing effect exists which leads to the hypothesis that the T_g of the film respectively plasticized polymer is close to the needed film formation temperature.

In order to define the curing temperature needed for film formation the pellets were cured at eight different temperatures between 25°C and 95°C. Postulating that the film formation temperature is close to the glass transition temperature (T_g) of the film the T_g was determined by thermo mechanical analysis (TMA). Therefore the pellets were inserted into a sample holder which allows the investigation of the coated pellet instead of a cast film. By this exceptional setup the penetration of the sensor into the polymer layer can be determined in situ, namely on the pellet's surface. Additionally, film formation was characterized by scanning electron micrographs (SEM).

Investigating pellets cured at temperatures up to 55°C by TMA, the temperatures at which the sensor penetrates into the polymer layer, are between 37°C and 39°C. This is caused by poor film formation which can be observed looking at the SEMs (fig. 1). During the measurement the porous structure of pellets cured at lower temperatures may collapse due to the rise of temperature which enables the TMA sensor to penetrate prematurely detecting a lower and consequently incorrect T_g. The temperature of penetration obtained from pellets cured at 65°C and above is between 43°C and 51°C. Looking at the SEMs (fig. 1) no porous structures are observed anymore. In order to measure the T_g and not the film's porosity of the lower cured pellets the T_g was redetermined after a second curing phase at 95°C for one hour expecting complete coalescence. All samples show T_gs between 46°C and 52°C indicating that a second thermal treatment leads to complete coalescence. A similarity to the T_gs obtained from the pellets cured at 65°C and above after the first curing phase is given.

Considering the results it can be shown that the T_g is a key parameter of the film formation of the dry coating process. Curing dry coated pellets at temperatures above the T_g leads to the coalescence of the polymer particle and formation of a functional film.

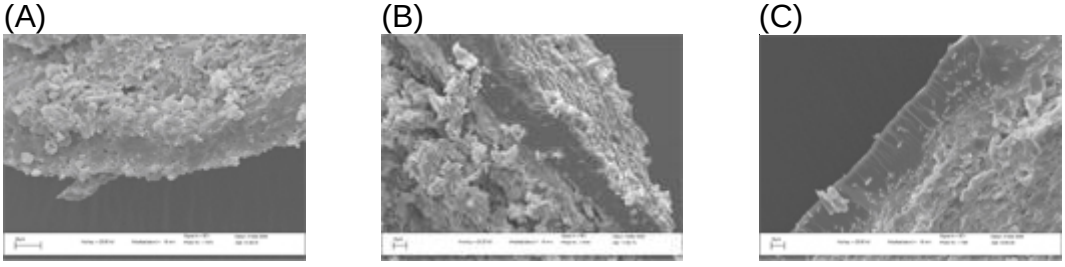

Fig. 1: SEMs of pellets cured at 25°C (A), 55°C (B) and 75°C (C), magnification 500x

(1) C. D. Kablitz, K. Harder, and N. A. Urbanetz. Dry coating in a rotary fluid bed, Eur. J. Pharm. Sci., 27, 212-219 (2006)