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Understanding the dissolution of a mineral material in formic acid

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Summary

A new inorganic binder for industrial use has been developed. The adhesive is based on sol-gel technology where the major component of the raw material is silica. The amorphous mineral material is dissolved in formic acid and gelation occurs after phase separation. The first part of the process, i.e. the solid-liquid dissolution reaction was investigated. The amount of undissolved material was weighed after phase separation and drying, in order to determine the amount dissolved. The conductivity and temperature were measured online. SEM-EDX and microscopic images were used to analyse the material before and after the reaction. Particle size distribution measurements were performed with laser diffraction. The dissolution rate increased with temperature as well as acid concentration. The particle size distribution did not vary significantly during the reaction. This observation was supported by the SEM and microscopic images taken from fresh and partly dissolved particles, which showed that holes and “craters” were formed on the initially smooth particle surfaces, but the overall dimensions of the particles was generally maintained. The nitrogen physisorption measurements indicated a significant increase in the surface area during reaction. Based on the experimental results, it is suggested that the material becomes more porous during the reaction maintaining its radial dimensions fairly well, followed by a rapid collapse of the silica skeleton. A mathematical model for the dissolution was developed. The conductivity can be used as a robust tool for measuring the progress of the reaction.

Key words: Dissolution, Particle size, Surface area, Conductivity, Kinetic modelling

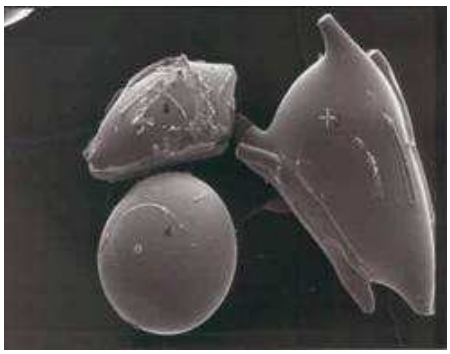
Extended abstract

A new low cost inorganic binder system for large volume products like fibre insulation, building materials, etc. has been developed based on sol-gel technology [1]. The precursor for the binder system is an amorphous mineral raw material containing silica as the major component. The sol was prepared by dissolving the amorphous mineral material in formic acid and the mineral was dissolved in a few hours depending on the molarity and temperature of the formic acid. The derived binder shows good wetting properties to mineral fibre surfaces and a good strength of paper-binder composites.

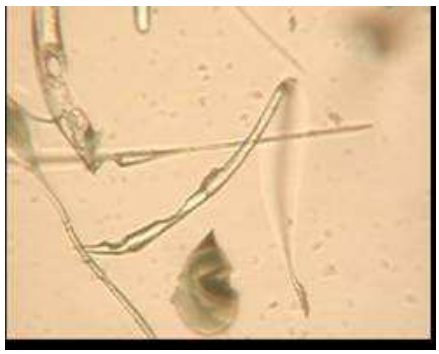
Experiments with dispersed particles were performed in an isothermal glass reactor at temperatures 288-313 K. Formic acid solutions between 0.6 and 3.75 M were used as the solvent. The conductivity and temperature were measured online. SEM-EDX and microscopic images were used to analyse the material before and after the reaction. Particle size distribution measurements were performed with laser diffraction. The change in the surface area of the particles during the reaction is investigated with nitrogen physisorption.

The dissolution rate increased with temperature as well as acid concentration. Elevating the agitation did not influence the reaction rate and thus, it was concluded that the agitation was sufficient, in order to reach the kinetic regime. The conductivity was influenced by the concentration of the dissolved matter and formic acid as well as the temperature. A phenomenological correlation between conductivity and concentration was established. This correlation can be used as a robust tool in industrial on-line determination of the amount dissolved.

The SEM and microscopic images taken of the fresh particles showed that there is a bimodal shape distribution of both spherical and elongated cylindrical particles present in the solid material. The images showed that the surface of the particles is smooth prior to the reaction. The images taken of partly dissolved particles revealed that holes or “craters” were formed on the surface, while the overall shape was generally maintained (Pictures 1 and 2).



Picture 1. SEM image of fresh particles of spherical nature.



Picture 2. A microscopic image of partly dissolved particles.

Laser diffraction measurements show that the particle size is maintained during the reaction (Figure 1), while the amount of dispersed particles declined. This suggests that the silica skeleton of the mineral material maintains its radial dimensions fairly well during dissolution and that the particles become more porous until they disintegrate dissolving rapidly. The observations made with SEM and microscope also support this behaviour.

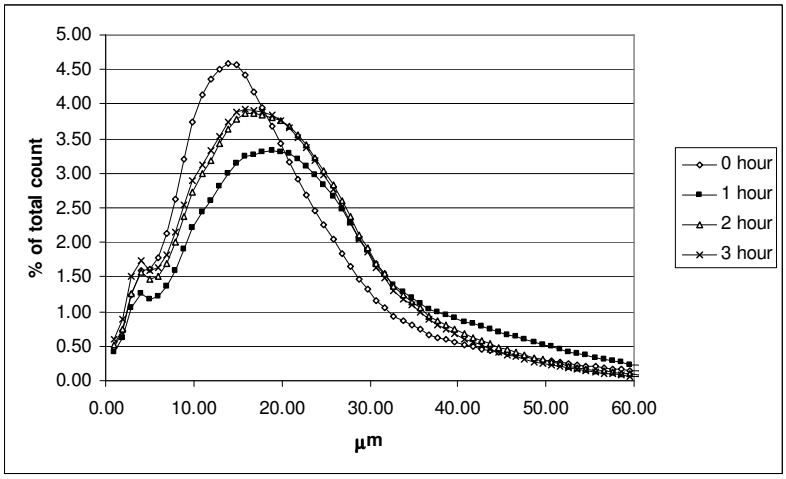


Figure 1. The particle size distribution of fresh and partly dissolved solid particles. The dissolution time of the experiments varied from 0 to 180 minutes. All the experiments were performed at 25°C and 2.5 M formic acid.

The nitrogen physisorption measurements indicated a significant increase, from around 1.5 m^2/g to over 5 m^2/g, in the surface area of the solid particles during reaction. Being indicative, these results support the observations made with SEM, microscope and laser diffraction measurements.

A model for the solid-liquid dissolution reaction was developed for use in designing pilot and industrial scale production. The effect of temperature and concentration as a function of time as well as the morphology of the solid particles is considered. The conductivity is utilized as a robust tool for measuring the progress of the reaction.

References

[1] Nilsen, E., Einarsrud, M.-A., Puputti, J., Lindén, M., Le Bell, J., Perander, M., (2005) *Journal of Sol-Gel Science and Technology*, 35, 143-150.