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THE INFLUENCE OF PRECIPITATION TEMPERATURE ON SILICA MORFOLOGY

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ABSTRACT
The precipitation temperature dependance of the morphology of silica produced via sodium silicate had been investigated in this article. Precipitated silica of highly dispersion degree was produced using two stage neutralization of sodium silicate solution. The influence of temperature on silica properties has been established on the basis of nitrogen gas absorption, dibutyl phthalate (DBP) absorption and SEM analysis. The results show that silica morphology largely depends on this process parameter.
Keywords: precipitated silica, morphology, precipitation temperature

1. INTRODUCTION

Reinforcing fillers are frequently used to modify the physical properties of polymeric materials. The term reinforcement is used to refer to enhancement of modulus, tensile strength, or wear resistance. Reinforcement of an elastomer depends on primary particles diameter, structure and surface activity of filler particles. Smaller particles have large surface areas, resulting in higher area available for interaction with the polymer. The reinforcing properties of silica are greatly influenced by its structure, [1]. Fillers are made up of primary particles at the smallest size-scale, Angstrom to micron, which are strongly bonded to other primary particles to form an aggregated structure. The aggregates range from the nanometer to micron size-range and these can interact with other aggregates through weaker secondary complex bonding to form agglomerates, [2,3]. Precipitated silica used for rubber reinforcement is usually synthesized through the controlled neutralization of sodium silicate (Na2O)(SiO2)3.3, with sulfuric, carbonic or hydrochloric acid. Precipitation produces a low solids content slurry of hydrated silica and residual salts, either sodium sulfate, sodium chloride or sodium carbonate. The salts are removed by washing in either a counter-current decantation system or by filter press. Drying is the last operation in production of silica, [4].

2. SYNTHESIS OF PRECIPITATED SILICA

Precipitated silica of highly dispersion degree was produced using two stage neutralization of sodium silicate solution. In the initial stage, the sodium silicate solutions were carbonized, and in the second stage acidification were continued with introduction of hydrochloric acid into solution until pH value of solution dropped to 4. Precipitation parameters: final pH value and reactant concentrations, were kept the same for all specimens, while the temperature changed. Precipitation conditions for all silicas produced are listed in table 1.

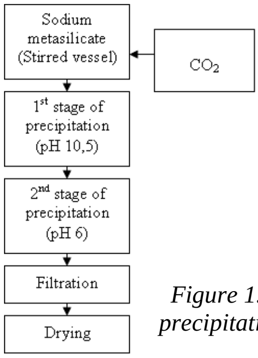

Figure 1. Two-stage precipitation of silica

Table 1. Precipitation conditions

Temperature [°]	20	40	60	80
SiO_2 concentration [%]	8	8	8	8
pH value	6	6	6	6

3. CHARACTERIZATION OF PRECIPITATED SILICA MORPHOLOGY

Reinforcement of an elastomer depends on primary particles diameter, structure and surface activity of filler particles. Smaller particles have large surface areas, resulting in higher area available for interaction with the polymer. Colloidal fillers, such as silica, exhibit structures over 5-7 decades in length scale and hence require a range of characterization techniques. The effect of increasing temperature on silica properties has been established on the basis of nitrogen gas absorption, dibutyl phthalate absorption and SEM analysis. In general, measuring the specific surface area has been a standard practice in studying filled rubbers. This properties is important for fillers because the structures vary widely at small sizes that are measurable by gas adsorption. Surface area was determined by N_2 adsorption method (BET) and the results are given in table 2 and figure 2. The bulkiness of aggregates is often referred to the "structure". It can be assessed by measuring the volume of dibutylphthalate (DBP) absorbed by a given amount of silica. High structure implies extensive bulkiness (porosity) and hence a low bulk density and great capacity to absorb Dibutyl pthalate (DBP). The results of silica DBP absorption are presented also in table 2 and figure 2.

Table 2. BET specific surface area and DBP absorption of precipitated silica specimens

Precipitation temperature [°C]	20	40	60	80
BET specific surface (m^2/g)	435,03	568,17	423,72	366,37
DBP absorption [ml/g]	1,45	2,69	2,45	1,60

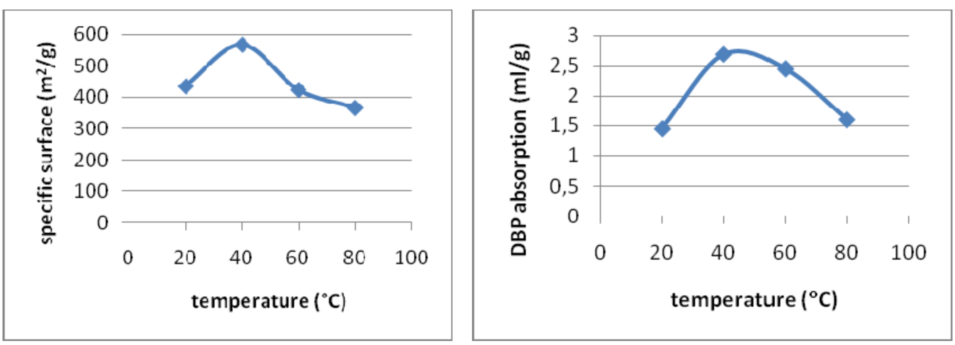

Figure 2. Influence of precipitation temperature on silica's specific surface and DBP absorption

It can be seen from figure 2 that silica produced at 40 °C has the highest values of specific surface area and DBP absorption. Decreasing or increasing precipitation temperature results in decreasing of specific surface area and DBP absorption of silica.

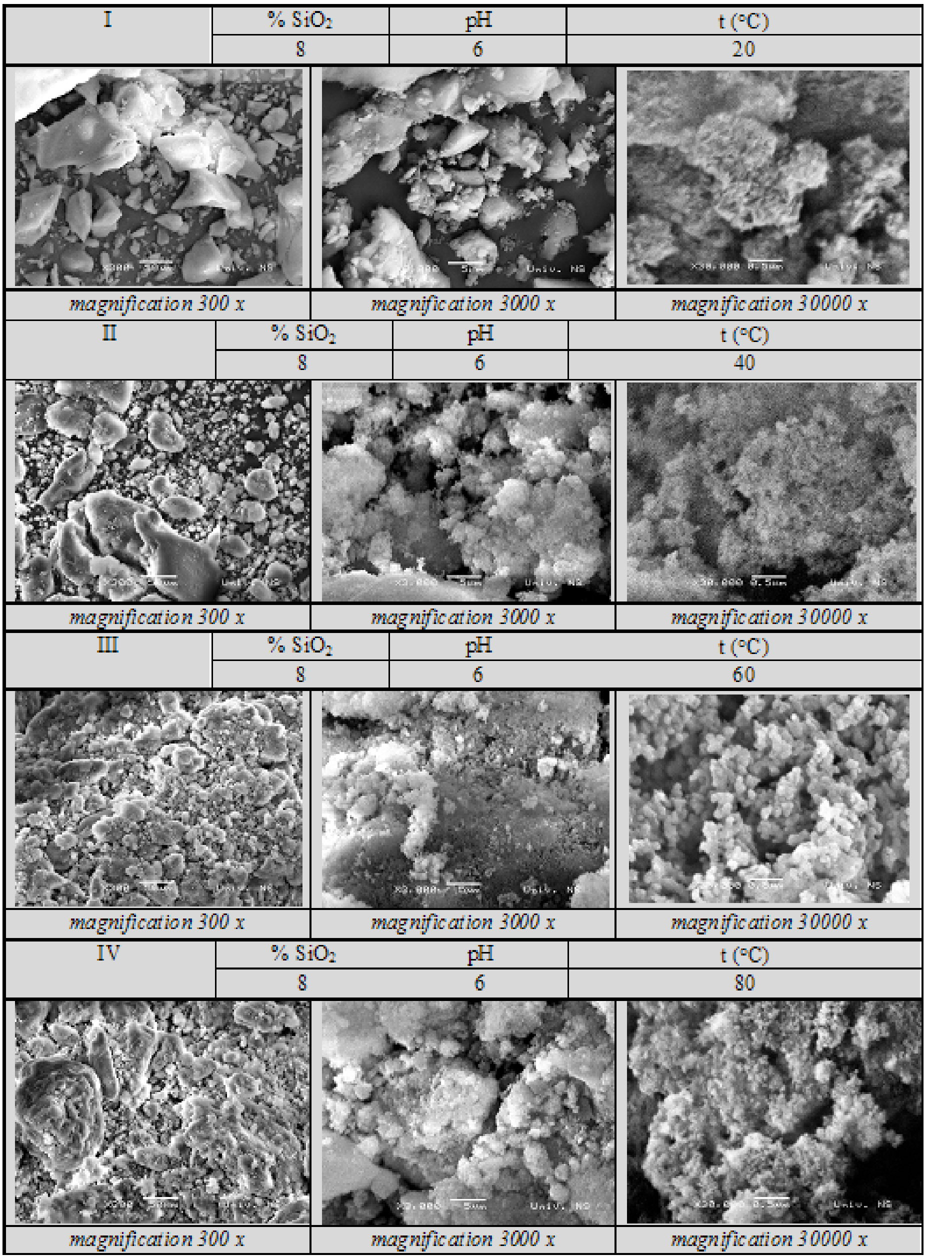

Figure 3. The influence of precipitation temperature on silica morphology

Scanning electron microscopy (SEM) is employed to investigate silica's surface morphology. The measurement is carried out by JEOL-JSM-6460LV microscope. SEM micrographs of silicas produced at temperatures: 20, 40, 60 and 80°C, are shown in fig. 32, in order to determine how precipitation temperature influences silica's morphology. To better detect differences in the structure, for each sample micrographs were taken at three different magnifications: 300, 3000 and 30000. The micrographs reveal the surface of the agglomerates, but not the size of the agglomerates. Also, the size of primary particles cannot be measured on these micrographs. But, it is possible to identify primary particles on micrographs with the greatest magnification (30000x).

4. CONCLUSIONS

The results presented show that precipitation temperature has a great impact on silica morphology. SEM micrographs reveal differences in all morphology levels of silicas produced at different temperatures. Also, precipitation temperature influences silicas specific surface area and DBP absorption. Micrographs show that silica produced at 60°C has the greatest primary particles. Any precipitation temperature change (increasing or decreasing) results in decreasing of primary particles size. However, silica precipitated at 20 °C has tendency of grouping primary particles into dense aggregates.

5. REFERENCES

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