doi:10.1017/S1431927613010210
Microsc. Microanal. 19 (Suppl 2), 2013

Study of Hydrated Lime in Environmental Scanning Electron Microscopy.

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Lime is an original binder for mortars for walling and plastering used during the millennia. Burnt lime (CaO) is hydrated and forms water suspension $CaO + H_2O \rightarrow Ca(OH)_2$ called lime putty. The process of hydration of calcium oxide and the age of lime putty stored significantly influences the technological properties of lime. Calcium hydroxide (portlandite) forms a hexagonal crystal in cross-section. Reducing the size of portlandite crystal is achieved through the aging of lime putty. In water suspension, calcium hydroxide crystals merge into hydrogel which affects the plasticity of fresh lime mortars. Crystals of portlandite absorb a large amount of water, and lime putty becomes excellently workable with good water retention and plasticity. In the view of the fact that the wet state of the sample is a natural part of the manufacturing process, the study of a sample free of dehydration and treatments is the most suitable for electron microscopy.

Environmental scanning electron microscopy (ESEM) is a widely used method that allows the overcoming of the limitations of the conventional SEM in order to facilitate the research and characterization of non-conductive, wet or liquid samples in particular. In the ESEM, samples can be observed across a wide range of sample temperatures, pressures of various gases (from units to thousands of Pa). etc., or in specific chemical conditions. In appropriate working conditions, wet non-conductive samples like hydrated CaO were observed in their native state without prior dehydration or other preparation for charging artifact elimination [1].

$Ca(OH)_2$ suspensions were prepared using reboiled deionized water. Fresh putties were immediately filled into plastic bottles, complemented with water to prevent drying; thereafter the samples were cured at room temperature till the test ages. Finally, sample washing in deionized water was applied for microscope contamination prevention. Samples of continuously aged $Ca(OH)_2$ were observed in a wet state using our experimental ESEM AQUAEM II [2] in conditions of a water-vapour environment with the pressure 750-850 Pa (5.6-6.4 torr) and sample temperature approx. 2.5°C. These settings allow the showing of the natural in situ structures of the $Ca(OH)_2$.

The long term study of the $Ca(OH)_2$ putty was shown in Figure 1. The sample of $Ca(OH)_2$ observed after 3 days, see Figure 1 A, shows good matured crystals of calcium hydroxide. The process of hydrogel formation is apparent from Figure 1 B when most of the hexagonal crystals of portlandite are coated with the shapeless columns. In Figure 1 C there is a structure with no observable crystals of $Ca(OH)_2$. The results showed a sequential transformation of portlandite crystals into calcium hydroxide hydrogel. From the viewpoint of lime putty utilization in mortars it is necessary to age it in water suspension for 6 months. Then all crystals are coated with hydrogel which causes a good plasticity of mortar.

References:

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[3] This work was supported by the Grant Agency of the Czech Republic: grant No. GAP 102/10/1410, by the EU projects No. CZ.1.05/2.1.00/01.0017 and No. CZ.1.05/2.1.00/01.0014.

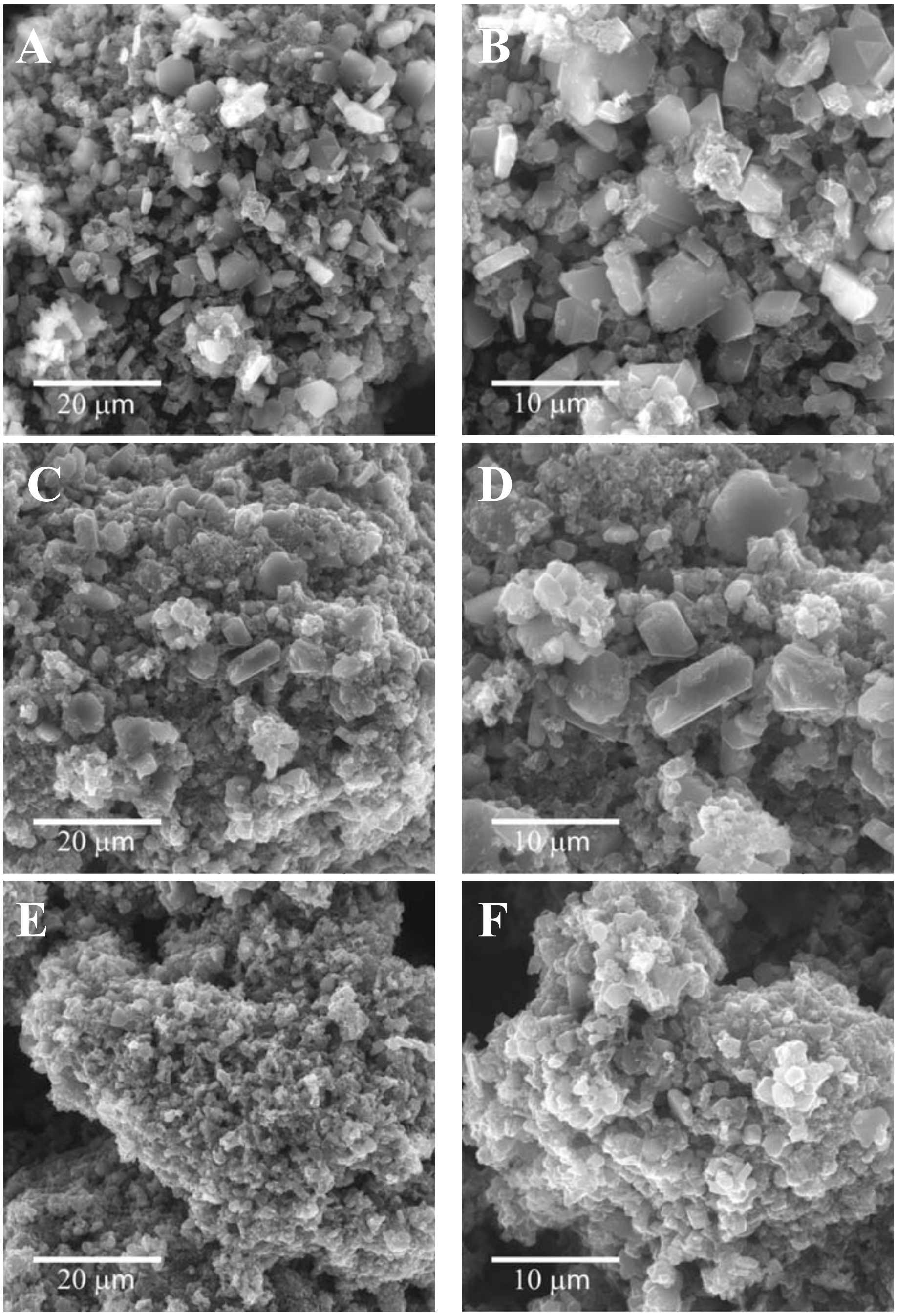

Figure 1. Aged $Ca(OH)_2$ putty A,B) after 3 days, C,D) after 36 days and E,F) after 183 days. Portlandite crystal size is gradually reduced as crystals are coated with hydrogel.

https://doi.org/10.1017/S1431927613010210 Published online by Cambridge University Press