Chapter V

Discussion

5.1 The Relation Between Particle Size and Specific Surface Area of Ash

Generally, for a powder material, particle size is the key parameter to determine the surface area. In rice husk ash, the geometric surface area, Γ, brought about by the secondaries can be calculated from their density ($\rho = 2.2$ g/cm^3) and size distribution by

$$\Gamma = [6/(\rho \cdot d_{50})] \cdot \exp(s^2/2) \qquad ,$$

yielding 112 m^2/g. This is the approximate value of non-treated samples, while the highest specific surface area (N$_2$) reaches more than 200 m^2/g. To compare this material with two types of commercial products: precipitated silica reaches Γ up to 800 m^2/g with globular agglomerates, and fumed silica is in the range of 50 - 350 m^2/g with fractal structure. It is assumed that the size of the secondaries of rice husk ash does not become finer during an acid treatment (see figure 6). Hence, curve A in figure 24 is considered typical of all investigated samples. TEM micrograph (figure 25 a) shows the tendency to form both globularly agglomerated and fractal structure. So, silica from rice husk ash takes an intermediate position between the two types of commercial products. The significantly higher BET surface of properly treated samples (surface enlargement factor 2) is attributed to the primary structure not resolved by TEM. The surface enlargement factor indicates that the

primaries have quite high coordination numbers, since otherwise a significantly higher factor would result. Treatment determines how much of the primary structure is still accessable to nitrogen after incineration.

5.2 Comparative Properties Between Silica from Rice Husk Ash and Commercial Silica (Fumed Silica Aerosil OX50)

In this thesis, amorphous fumed silica aerosil OX50 from Degussa was selected to be compared with rice husk ash based on the same range of particle size distribution of both material. The general properties, purity, particle size distribution and particle size, surface area, and nature of agglomerate were compared, as shown here.

Tab. 13. Comparison data between silica from rice husk ash and aerosil OX50

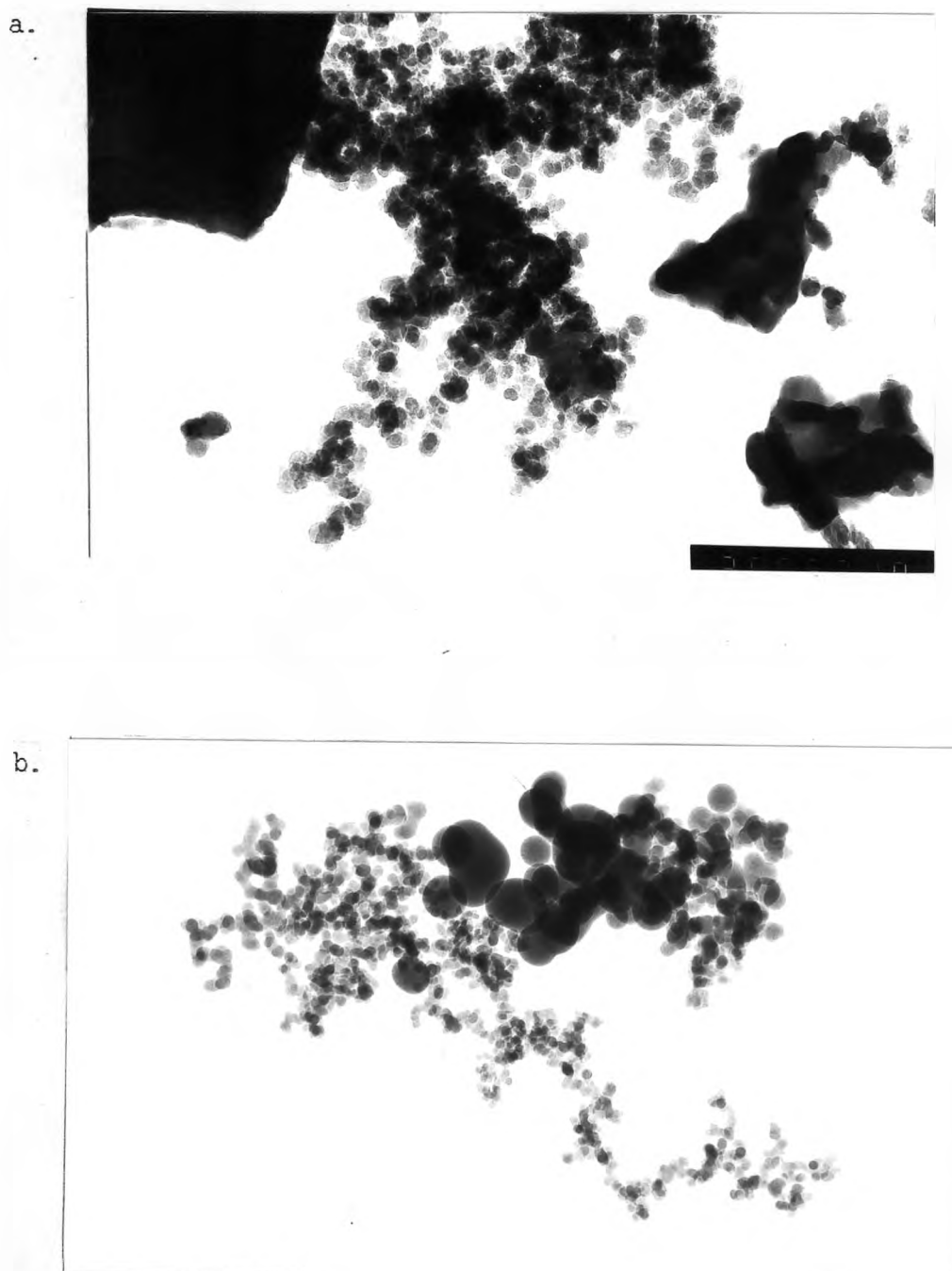| Property	Rice husk ash	Aerosil OX50
purity (% SiO_2)	> 99.4	> 99.8
d_0, particle size, primary;	2.4 nm	40 nm
secondary	26 nm	–
surface area	> 200 m^2/g	50±15 m^2/g
nature of agglomerate	between globular and fractal agglomerate	fractal agglomerate
surface behavior	hydrophilic	hydrophilic

a.

b.

Fig. 25 a-b TEM micrographs of rice husk ash and aerosil OX50

a: rice husk ash; b: aerosil OX50

|———————| · a: 330 nm; b: 500 nm

a.

20KV X5000   2233   1.0U   A
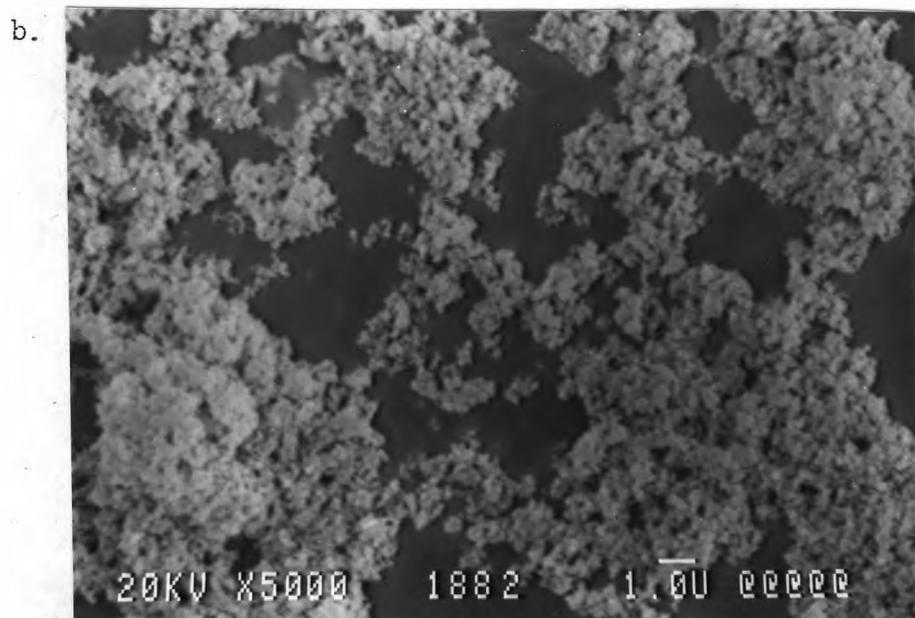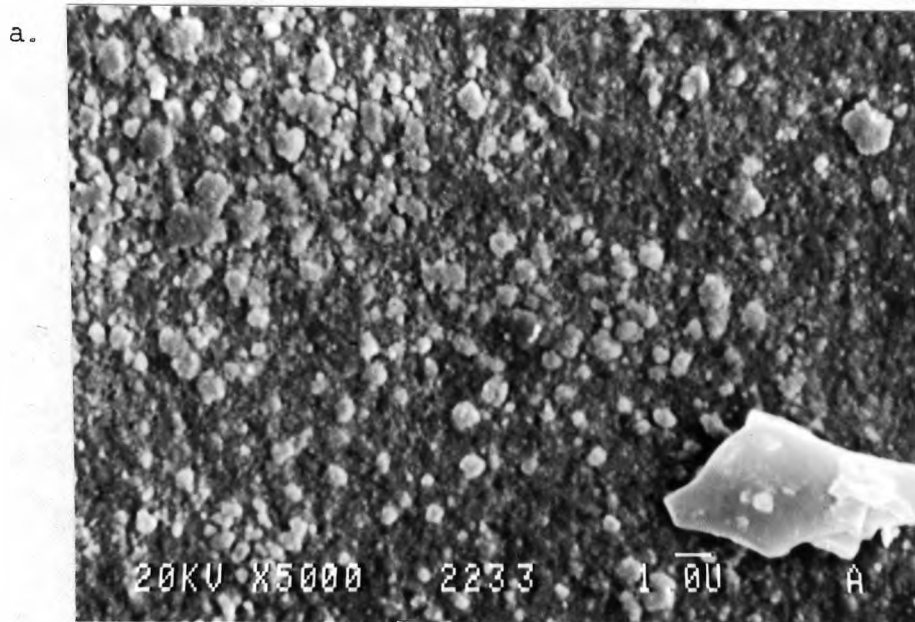
b.

20KV X5000   1882   1.0U eeeee

Fig. 26 a-b SEM micrographs of rice husk ash and aerosil OX50

a: rice husk ash; b: aerosil OX50

⊢⊣ 1 μm

The SEM micrographs show that aerosil OX50 has excellent dispersion properties (sample preparation of both material on SEM and TEM are the same) with agglomerate size lower than 0.5 μm. Rice husk ash can be prepared with a sub-micron agglomerates, but it has not so good dispersion properties. In the TEM micrographs, aerosil OX50 looks like solid spheres with a high degree of fractal agglomeration, while rice husk ash has irregular particles looking like porous small grains. Although commercial silica (aerosil OX50) is an easily dispersable material, it has a lower specific surface area. This means that the structure of aerosil OX50 as shown by TEM is the primary structure already, while for rice husk ash, there is still a lower level of structural units which cannot be dissolved by our TEM. To summarized: Due to its excellent dispersion properties, aerosil OX50 is the superior quality. But rice husk ash is not too far from reaching an acceptable standard.

5.3 Problem and Next Steps

In the following, some remaining problems and their potential solution are sketched.

During rice husk treatment, large amounts of water and mineral acid were used. This brings up the problem of waste water treatment, which is absolutely required if any up-scaling is envisaged. A potential and partial solution is the improvement of enzymatic treatment, which requires pH $\simeq$ 4 only. Besides this, the leachates contain valuable substances such as lignin which can be made available for further utilization. A co-operative effort between material scientists, chemists, microbiologists, and chemical technologists

would be the appropriate answer to establish suited recycling processes.

Although the silica product has already excellent properties as far as purity, specific surface, and nanostructure are concerned, the dispersion of the agglomerates is still unsatisfactory. This requires a strong focus on the powder-technological side of the problem. There are no principle objections towards reaching a product with agglomerates < 3 μm (Cremer, 1988.). This latter point is decisive for the future range of application of the silica product.

Characterization must go beyond what was done during the present thesis. One of the main shortcomings is the characterization of crystallinity. It is by far insufficient to use results from X-ray diffraction as a criterion for a nonastructured powder. Small angle X-ray scattering and high resolution transmission electron microscopy (both not available during the thesis work) are recommended. Beside this, information is required on the state of nucleation during heat treament, e.g., by establishing so-called TTT (temperature-time-transformation) diagrams for the product.