

Example 2

To produce an aqueous HPMCAS dispersion, water was loaded first and recirculated through a Drais DCP-12 media mill (1.0 mm Ytterium Stabilized Zirconia media, 0.5 mm screen size). During the milling process HPMCAS solids were loaded gradually to water having a temperature of 16-20 °C and recirculating through the mill at a mill speed of 1000 rev/min. Addition of HPMCAS was continued until a total solids loading of about 20 % was achieved, based on the total amount of the composition. At this point, 20 wt.% NH_4HCO_3 was added to the mill to adjust the pH to 4.7. Milling continued until the final median particle size, d50, of 1.1 μm was obtained.

The resultant degree of neutralization of succinoyl groups was 28 mole percent, i.e., 28 mole percent of the groups - $\text{CO-CH}_2\text{-CH}_2\text{-COOH}$ of the HPMCAS comprised in the aqueous dispersion were neutralized and present as - $\text{CO-CH}_2\text{-CH}_2\text{-COO}^- \text{NH}_4^+$ groups. The resultant dispersion had a solids content of 20.2 weight percent and a pH of 4.7. The HPMCAS particles had a median particle size, d50, of 1.1 μm , a mean particle size of 1.6 μm and a particle size d90 of 3.4 μm .

The freshly prepared HPMCAS dispersion had a good fluidity. The HPMCAS dispersion gradually lost some of its fluidity upon standing during aging at room temperature for a time period of 22 days, but the HPMCAS particles could easily be re-dispersed in the aqueous diluent by simple, low intensity stirring.

Comparative Example B

The procedure in Example 2 was repeated, except that ammonium hydroxide was added as a 15 wt. % ammonium hydroxide solution to the mill to adjust the pH to 4.8.

The resultant degree of neutralization of succinoyl groups was 33 mol percent, i.e., 33 mole percent of the groups - $\text{CO-CH}_2\text{-CH}_2\text{-COOH}$ of the HPMCAS comprised in the aqueous dispersion were neutralized and present as - $\text{CO-CH}_2\text{-CH}_2\text{-COO}^- \text{NH}_4^+$ groups. The resultant dispersion had a solids content of 20.2 weight percent and a pH of 4.8. The HPMCAS particles had a median particle size, d50, of 1.6 μm , a mean particle size of 2.0 μm and a particle size d90 of 4.0 μm .

The freshly prepared HPMCAS dispersion had a reasonably good fluidity, although it had a higher viscosity over the temperature range of 2 to 72 °C than the freshly prepared HPMCAS dispersion of Example 2. The HPMCAS dispersion gradually lost its fluidity during aging at room temperature for a time period of 22 days. Severe aggregation of solid particles and a hard crust were observed after 22 days. A meaningful viscosity measurement was not possible after 22 days. After an effort to re-disperse the HPMCAS particles, still some aggregated particles of increased density were visible that could not be re-dispersed into an apparently homogeneous dispersion.

For assessing the storage stability of the dispersions, the particle size distributions in diluted dispersions of Example 2 and Comparative Example B were re-measured after their preparation as listed in the table below by laser diffraction particle size analysis as described in the paragraph “HPMCAS particle size measurement in the aqueous dispersion” at page 22 of the patent application. The results are listed in the Table below.

(Comp.) Example	Days after preparation	Mean (µm)	d50 (µm)	d90 (µm)
2	31	8	2	29
2	55	13	3	30
B	24	6	2	20
B	48	37	6	140

The results in the Table above illustrate that the HPMCAS of Example 2, which is partially neutralized with NH_4HCO_3 , has much better storage stability than the HPMCAS of Comparative Example B which is partially neutralized with ammonium hydroxide.