

Effect of method of preparation of coating colors on the rheological behavior and properties of coating layers and coated papers

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THE MINERAL-BASED SUSPENSIONS used to coat paper and board are complex systems because they are concentrated and contain several different components. The components of the suspension or coating color interact with each other. This affects the structure and properties of the system. For instance, these interactions can influence the rheological properties of the coating color (1). Changes in the rheological behavior may have consequences on the runnability of the color. In principle, an aggregation of the pigment particles induced by electrolytes or by the water-soluble polymers in the color can increase the porosity of the coating layer and improve its covering ability.

One consequence of the complexity of the coating colors or of the interaction between the components of the color is that the properties of the color—its viscosity—can depend on the order of mixing the different components and the length of the preparation time (2–5). The energy required for the preparation of the color may also be different depending on the preparation technique (6). Certain combinations of components or a certain order of mixing the color components can lead to a “viscosity shock” (6). The number of reported systematic investigations about the preparation method for the rheological properties of the suspensions is small, how-

ever.

The few reported studies concentrate on the rheological properties and on the power requirement for the preparation. There has only been limited investigation of the possible effects on the properties and structure of the coating layer and of the coated substrate. This was therefore the subject of the present study.

This work used two conventional types of coating colors based on kaolin clay with latex as binder. In one case, carboxymethyl cellulose (CMC) was the water-soluble polymer. In the other case, it was poly(vinyl alcohol) (PVA). The coating colors were prepared in different ways and their rheological behavior evaluated. The colors were then used to produce coating layers on polyester film and to coat paper in a high-speed laboratory coater. Properties of the coating layers—porosity and light-scattering efficiency—and of the coated papers—optical properties and gloss—were measured. The power requirement during the preparation of the coating colors was followed with a technique similar to that used by Mäkinen and Järvensu (6).

EXPERIMENTAL

Materials

All pigments and chemicals were commercial grades used as received without further purification. An English coating clay grade (Speswhite from English China Clay Interna-

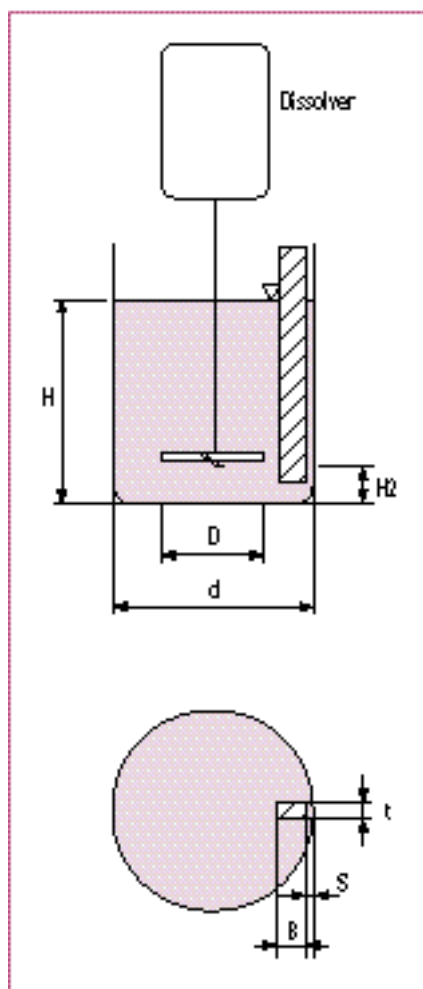
ABSTRACT

Due to the interactions between the components of a coating color, the manner of color preparation can affect the properties and structure of the suspensions and the coating layer. This work investigated two coating colors of clay with latex binder using either carboxymethyl cellulose or poly(vinyl alcohol). Certain mixing orders gave a significant increase in the shear viscosity and/or viscoelastic parameters characterizing the colors. This suggested a flocculation or aggregation of the coating color. In drawdowns on film, the orders of mixing the components that apparently resulted in a flocculation of the colors also increased the porosity of the coating layers and enhanced their light scattering efficiency. When these colors were used to coat a woodfree paper using a high-speed laboratory coater, a relatively marked increase in the gloss level (after calendering) was noted. The positive effects of changing the preparation procedure gave, in this case, however only a rather limited improvement in brightness and opacity of the coated paper.

Application:

The method of mixing of coating colors can influence flocculation leading to increased porosity and improved light scattering efficiency of coating layers on polyester films.

tional Ltd., England) was the pigment in all coatings and suspensions. According to the manufacturer, 80% of the clay particles had an equivalent spherical diameter less than 2 μm , and the specific surface area according to the Brunauer, Emmett and Teller (BET) method was 10.5 m^2/g . Sodium polyacrylate (NaPA, Dispex N 40 from Allied Colloids, England) was the dispersant for the pigment. The molecular weight was approximately 3000 according to the manufacturer. Sodium hydroxide



1. Schematic picture of the vessel used for the preparation of the coating colors with $H = 110$ mm, $H_2 = 25$ mm, $D = 60$ mm, $d = 130$ mm, $B = 15$ mm, $t = 4$ mm, and $s = 3$ mm

was also used when dispersing the mineral particles.

The binder was a latex based on a styrene butadiene copolymer (carboxylated latex, DL 685, from Dow Rheinmünster GmbH, Germany). The particle diameter was approximately $0.15\ \mu\text{m}$ according to the manufacturer. Glass transition temperature of the polymer was 25°C as determined by a torsional pendulum technique (7).

CMC with a low molecular weight (Finnfix 5 from Metsä-Serla Chemicals OY, Finland) was the cobinder. According to the manufac-

turer, the degree of substitution was 0.72. In a series of experiments, PVA was the water-soluble polymer in the suspensions. The PVA grade (Mowiol 10-98, Hoechst AG, Frankfurt am Main, Germany) had a degree of hydrolysis of 98% and a molecular weight of 49,000 according to the manufacturer.

Preparation of coating colors

Kaolin-based coating colors were prepared in five different manners described below. The amounts of the different components were 0.3 parts by weight of the NaPA per 100 parts (pph) of kaolin; 0.15 pph NaOH (in most cases), 1 pph CMC, 1 pph PVA, and 10 pph latex. The different components were not all used simultaneously in a given coating color.

Power requirement during the color preparation

The power requirement was measured as a function of the preparation time with a laboratory dissolver (Dispermat CV from VMA-Getzmann, Germany). The power requirement during one minute was measured 25 times and represented by the average value during that time. Measured values included any power losses in the dissolver. The different components were mixed with a dissolver disc having a diameter of 60 mm. The vessel used for the preparation of the coating colors had one baffle as Fig. 1 shows.

Rheological properties of the suspensions

The flow curves in shear of the suspensions up to a shear rate of $1460\ \text{s}^{-1}$ were measured at room temperature ($23 \pm 0.5^\circ\text{C}$) using a Bohlin VOR rotational viscometer (Bohlin Reologi AB, Sweden) with a Couette configuration. The viscoelastic properties of the colors were evaluated in shear oscillation mode at frequencies between 0.02–10 Hz. Characterization used a storage G' and a loss modulus G'' . The dynamic mechanical measurements were made in the linear viscoelastic region—the

applied shear amplitude was sufficiently small not to affect the values of the moduli.

Preparation of coating layers

The coating colors were drawn down on an impervious polyester film (Mylar, Du Pont, USA) with a thickness of $100\ \mu\text{m}$ using wire wound rods. The coating layers were dried at room temperature, and the dry coating weight varied between 11 – $54\ \text{g/m}^2$.

Laboratory coating of paper

A neutrally sized woodfree base paper (from MoDo Husum Paper AB) with a grammage of $70\ \text{g/m}^2$ was coated on one side with the different coating colors using a Helicoater (ECC International Ltd, England). The machine speed was $600\ \text{m/min}$, and the coating weight varied between 5 – $12\ \text{g/m}^2$. After the coating, the papers were conditioned at 23°C and 50% RH.

Thickness measurements

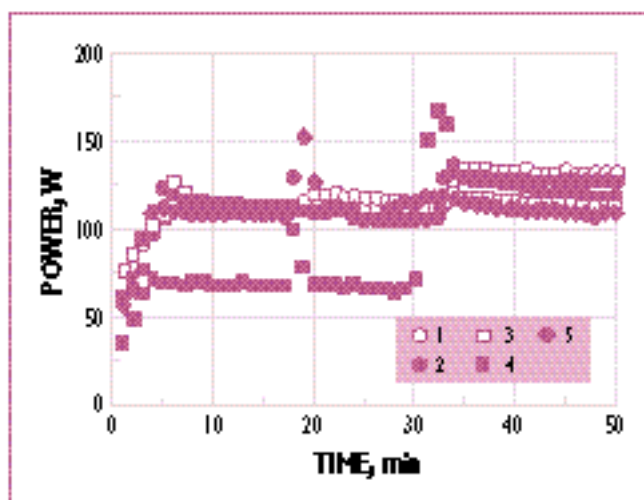
The thicknesses of the coated polyester films and of the coated papers were measured between spherical platens using a thickness tester developed at STFI, Stockholm, Sweden (8).

Determination of optical properties

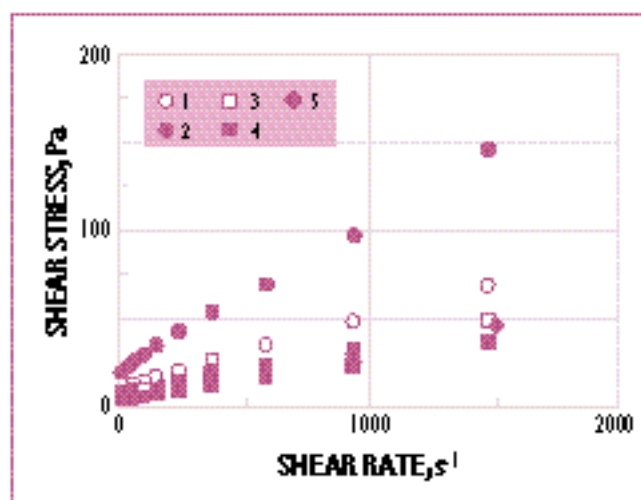
The light-scattering coefficient of the coating layers in the polyester film was calculated for different wavelengths between 400 – $700\ \text{nm}$ using the Kubelka-Munk theory from reflectance measurements made against white and black backgrounds with an Elrepho 2000 instrument. For the coated paper, the optical properties—brightness and opacity—were evaluated with an Elrephomat instrument using standard procedures.

Calendering of the coated papers

The coated papers were calendered twice in a paper to steel nip with a laboratory calender (Gebr. Kersten, Eerbeeksche Mach. Fabrik, the



2. The power requirement vs. the preparation time for the five different clay based coating colors



3. The shear stress as a function of the shear rate for the five different clay based coating colors with a solids content of 53.5%

Netherlands) using a line load of 155 kN/m. The rolls of the calender were not heated.

Gloss measurements

Gloss measurements in the coating direction on the coated and calendered papers used a Hunter gloss meter according to TAPPI T 480.

RESULTS

Power requirement for the color preparation

The coating colors were prepared at a rotational speed of 5000 rpm in five different ways:

1. Kaolin clay was dispersed in water containing 0.15 pph NaOH and 0.3 pph sodium polyacrylate. The starting point of the preparation was when kaolin was transferred to the liquid. After 17 min, 1 pph CMC was added. After 32 min, 10 pph latex was added to the suspension.
2. Kaolin was dispersed in water containing NaOH and CMC and after 32 min latex was added to the suspension. Here slightly more than 0.15 pph NaOH had to be used—corresponding to a pH of 10—to facilitate the incorporation of the latex into the color.

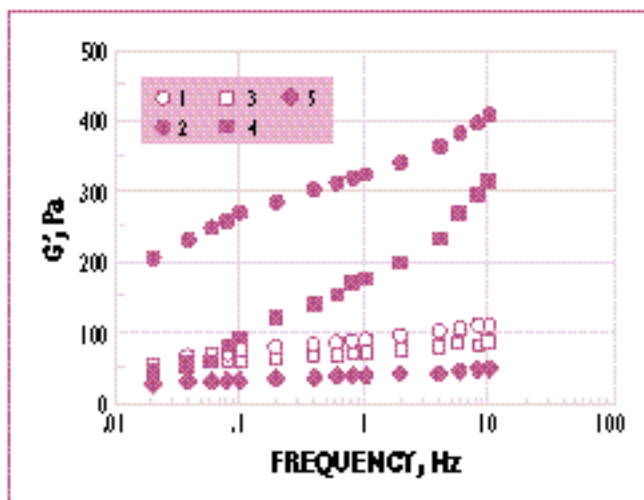
Note that CMC actually was used as a “dispersant.”

3. Kaolin was dispersed in water containing NaOH, sodium polyacrylate and 1 pph PVA. After 32 min, 10 pph latex was added to the suspension.
4. Two suspensions were prepared. The first was prepared by dispersing clay in water containing NaOH and sodium polyacrylate. For the second, the kaolin was dispersed in water containing NaOH and sodium polyacrylate and after 17 min, 1 pph PVA was added. Thirty minutes after the start of the preparation of the second suspension, equal amounts of the two suspensions were mixed, and latex was added after one minute. This color finally contained 0.5 parts of PVA per 100 parts of kaolin.
5. Kaolin was dispersed in water containing NaOH and sodium polyacrylate. PVA (1 pph) and latex (10 pph) were added to the suspension after 17 min and 32 min respectively.

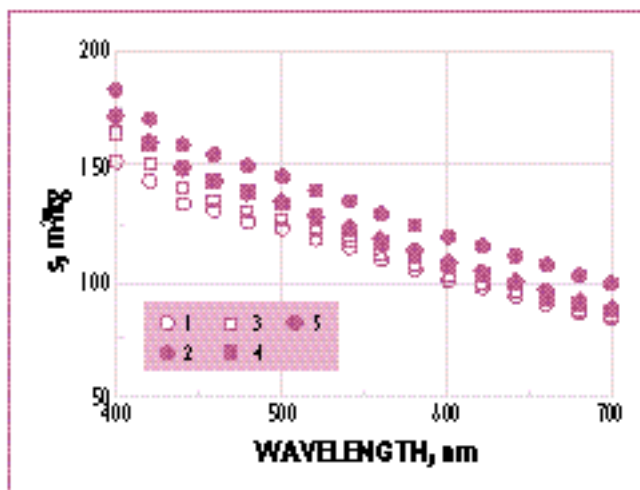
The different colors and corresponding coating layers in the following discussion have numbers as indi-

cated above. **Figure 2** shows how the power requirement varied during the preparation cycle for these systems. The final solids contents of the coating colors were the same in all cases—53.5%. This low solids content level was chosen to prepare all the different coating colors easily. Although the properties of the colors and the corresponding coating layers might not be identical to those obtained at a higher solids content, the results presented here illustrate important trends.

The power requirement increased when the pigment was dispersed in the aqueous phase. The power requirement increased further when CMC and latex were added to the suspensions. These increases are partly due to the corresponding increase in the mass in the vessel and to the increase in the viscosity of the system. When PVA was added, there was a significant rise in the power requirement resulting in a maximum of a short duration—colors 4 and 5. A similar maximum also developed when mixing the two suspensions constituting color 4. There was no maximum in the power requirement, however, when dispersing the kaolin in PVA containing liq-



4. The storage shear modulus, G' , for the five coating colors vs. the frequency



5. The light-scattering coefficient, s , for the different coated polyester films vs. the wavelength of light with a coating weight of approximately 11 g/m^2

uid—color 3. The low power requirement up to 30 min in the preparation of color 4 is simply because only half the final mass was agitated in the vessel. The final power requirement to agitate the colors—after 40–50 min preparation—was the same order of magnitude for all the colors.

Rheological properties of the coating colors

Figure 3 depicts the steady-state shear flow curves for the five colors. For all the colors, there was an approximately linear relation between the shear stress and the applied shear rate. Obviously, the manner in which the CMC containing colors were prepared influenced the flow curves appreciably. The highest shear stress at a given shear rate occurred when the kaolin pigment had been dispersed in the CMC solution without the NaPA dispersing agent—color 2. The PVA containing colors exhibited a similar flow behavior, however, in shear. The shear stress at a given shear rate was always lower than that noted for the CMC containing colors. There were lower shear stress values for color

4—the suspension with the lowest PVA content.

Figure 4 shows that the shear storage modulus G' increased with increasing frequency for all five colors. Color 2—kaolin dispersed in the CMC solution without any NaPA present—exhibited a substantially higher storage modulus than the other suspensions. When a PVA containing color was prepared using the two-stage method (color 4), high G' values occurred for frequencies exceeding approximately 0.5 Hz. This occurred although the amount of water-soluble polymer in this suspension (0.5 pph) was lower than in the other systems. The frequency dependence of G' was also stronger for this suspension than for the others. The lowest values of the storage modulus were noted with color 5—PVA and latex added to the dispersed clay suspension.

For all five coating colors, the loss modulus G'' was substantially lower than the storage modulus. This shows that these colors had a pronounced elastic character at low deformations over the frequency range investigated.

Porosity and light-scattering efficiency of the coating layers

Plotting the thickness of the coating layers on the polyester film as a function of the coating weight produced a linear relation for all the different systems. Table I shows the porosities of the coating layers evaluated from the slope of this line and the densities of the components of the coating colors.

The standard deviation of the measured porosities was typically 1% (4). Porosities of the coating layers were of an order of magnitude typical of this type of material (9). The difference in porosity between the layers is not substantial, but it is still significant. The highest porosities occurred for the systems that exhibited a deviating rheological behavior—colors 2 and 4.

Figure 5 shows the light-scattering coefficient, s , as a function of the wavelength of light for the five coating layers on the polyester film. The coating weight was approximately the same for all the layers at approximately 11 g/m^2 . The effect on the s value from the transparent polyester film should be almost negligible. The

s values in Fig. 5 are therefore approximately those of the coating layers themselves.

The light-scattering efficiency of the different layers appeared often to follow the same pattern as the porosity. The layers based on colors 2 and 4 had the highest s values. The layer based on color 1 exhibited the lowest s values and the lowest porosity. Note that there was a substantial difference in the s values between the two CMC containing layers—1 and 2—despite their similar composition. At the lowest coating weight used of 11 g/m² (Fig. 5), the s value of layer 5 was close to that of layer 4. They almost coincide in the graph. This was not the general behavior, however, since at the higher coating weights the s value of layer 5 was substantially lower and closer to that of layer 1. This is also more consistent with the porosity value of layer 5.

The same pattern in light-scattering efficiency also occurred at higher coating weights. Here the differences between the different layers became smaller. The magnitude of the s values also decreased as the coating weight increased.

Properties of the coated papers

The woodfree base paper could be coated using the Helicoater without any significant problems—there were no runnability problems.

Figure 6 shows how the brightness of the coated papers developed as the coating weight increased. The papers were not calendered. The uncoated and uncalendered base paper had a brightness of 86.8% and an opacity of 84.9%.

Despite the differences in the light-scattering coefficient among the coating layers on the polyester film in Fig. 5, there was virtually no difference between the brightness values of the coated papers. Calendering did not affect this result. It merely reduced the brightness by approximately 1%. The different

Color no.	Porosity, %
1	31.4
2	34.9
3	34.0
4	35.6
5	32.2

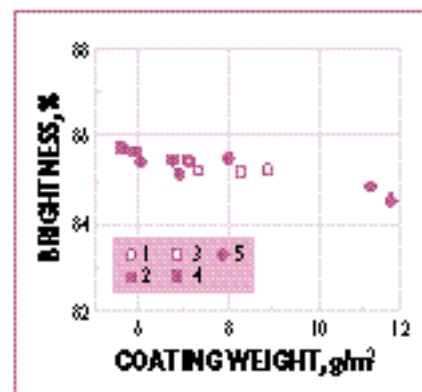
1. Porosities of the coating layers on the polyester film

methods used to prepare the colors obviously had no influence on the brightness of the coated papers here. They also did not substantially affect the opacity as Table II shows. The values here came from interpolation to a coating weight of 8 g/m². There was a small tendency for color 2 to give a higher value of the opacity. The lowest opacity occurred with color 1. The differences between the opacity values of the different papers are within the standard deviation of the measurements, however.

The gloss after calendering depended to some extent on the manner of color preparation. Higher gloss levels resulted when the wood-free paper was coated with colors 2 or 4 as Table III indicates. The standard deviation of the measured gloss values was approximately 1%.

DISCUSSION

The power requirement for the preparation of the colors was similar for the different systems used. The only major exception was when PVA was added to the suspensions. The power requirement then rose significantly and passed through a maximum. One can understand this in terms of a bridging mechanism (5, 10, 11). Particle collisions may bring uncovered areas of one particle into contact with areas of another particle covered with adsorbed PVA leading to bridging flocculation. The flow field during the preparation was efficient in breaking the aggregated structure, however. The short duration of the power maximum shows this. When the pigment was



6. The brightness of the papers coated with the different coating colors as a function of the applied coating weight for papers not calendered after the coating

dispersed in the PVA-containing phase instead of having the PVA added to the clay suspension, no maximum or “blending shock” occurred at the polymer dosages used here. The flocculation was suppressed at least partially. This resembles the results reported by Jayme and Schempp (12) in their studies on coating colors containing PVA. It is difficult, however, to obtain information regarding the structure of the suspensions from the power needed to agitate the final colors at longer times. Besides the flow field, the power requirement may also depend on the rheological properties of the system (13, 14). Those differ for the colors produced here.

The CMC and PVA containing colors have separate discussions at first here since they are two different systems. The two CMC containing colors differed appreciably in rheological behavior as Figs. 3 and 4 show. Color 1 was prepared consistent with the procedures used in practice today. The viscosity, the yield stress (the shear stress when extrapolating the flow curve to zero shear rate), and the storage modulus were substantially higher when the clay particles were dispersed in the CMC solution—color 2. The plastic viscosity evaluated from the slope of

Color no.	Opacity, % before calendering	Opacity, % after calendering
1	88.3	86.8
2	88.7	87.3
3	88.4	87.0
4	88.5	87.2
5	88.4	87.2

II. Opacity of the coated papers at a coating weight of 8 g/m²

the linear part of the flow curve was also higher for this color. In terms of the elastic floc model (15), this latter observation indicates a more flocculated structure of the suspension than in color 1.

One may speculate on the reasons for this difference. A suggestion is that CMC forms a network in the liquid phase of the coating color (1, 16). This is one reason for the comparatively high values of the storage modulus of such colors. This applies to colors dispersed with NaPA like color 1. Although a network may form in such systems, the adsorption of CMC onto the kaolin particles is not very significant (17). When the clay particles are dispersed without any NaPA, the adsorption of CMC onto the pigment particles is enhanced (17). Now it is possible to incorporate the clay particles into the polymer network to a greater extent. In effect, the suspension appears more aggregated. The plastic viscosity and the modulus increase correspondingly. It may also be possi-

ble that bridging flocculation can play a role when the clay suspension contains no dispersant. Note that the differences between color 1 and 2 reported here also show that NaPA is a powerful dispersion agent for clay. This is not very surprising. Järnström and Stenius have analyzed the interaction between NaPA and clay in greater detail (18).

The PVA containing color 4 prepared using the two-stage method also exhibited a high storage modulus indicative of a flocculated structure. A bridging mechanism as discussed in detail by Järnström *et al.* (4) can explain this. The PVA content of this color is not sufficient for complete coverage of the pigment by the polymer (4). This favors bridging between the clay particles—one polymer molecule may anchor to two particles. When the PVA content is sufficiently high as in colors 3 and 5, the adsorption leads to a complete coverage of the clay particles. The polymer then stabilizes the suspension (19). Then flocculation is suppressed.

The dynamic mechanical measurements were performed on a structure that to a first approximation was undisturbed at rest. The structure of color 4 then appeared quite stiff. This structure seems to break down, however, when the suspension is subjected to a shear flow as Fig. 3 indicates. The flow curve for this color does not differ substantially from those of the other two PVA suspensions. This may indicate that the flow elements during the

Color no.	Gloss, %
1	39
2	43
3	39
4	42
5	39

III. Gloss of the coated and calendered papers at a coating weight of 8 g/m²

shearing in color 4 are not aggregates to any great extent. After stopping the shear, the color again exhibited a high G' value indicating that the stiff structure recovers quickly. The rheological behavior of the PVA color 4 contrasts in some respect with that of the CMC color 2. The plastic viscosity of the CMC color increased significantly when the preparation technique changed. This suggests that for this system the flow elements during shearing are porous aggregates. The PVA content of color 4 was only half that in colors 3 and 5, however. The flow curves in Fig. 3 were nevertheless quite similar. These curves therefore also indicate a higher degree of aggregation in color 4 than in the other two PVA containing ones (4).

In principle, an aggregation of the pigment particles may increase the porosity of the dry coating layer and thus increase the possibility of improving the coverage of the substrate by the coating layer. This is important since a uniform and high covering ability is a prerequisite for obtaining a high print quality. The rheological behavior exhibited by color 4 may provide an advantage in this regard. The formed aggregate structure easily breaks down when shearing the suspension. This may promote runnability during the coating operation (20). After cessation of the flow, the aggregated structure recovers again. This can result in a higher porosity in the dry coating layer.

The results reported here sup-

KEYWORDS

Addition polymers, carboxyalkyl celluloses, carboxymethyl cellulose, cellulose ethers, clay, coated papers, coating color, coatings, kaolin, latex, minerals, paper grades, physical properties, polyvinyl alcohol, polyvinyls, preparation, process variables, properties, rheological properties, sequencing, thermoplastics, variables.

port the view that flocculation can increase the porosity of the coating layers as Table I shows. The highest porosities were observed in the coating layers based on colors 2 and 4. The higher porosity of layer 4 compared to layers 3 and 5 is noteworthy, since the former layer contained less PVA than the latter two. At the polymer levels used here, one would expect that a higher PVA content should give more expanded—more porous—structure (21). The flocculation of the clay particles obviously changes the situation.

Because of the large differences in rheological behavior between the coating colors, the differences in porosity may not appear very great. The aggregated structure in the wet state probably collapses to some extent during the consolidation of the coating layers as described by Larrondo and Lepoutre (22). This results in smaller differences in the dry state between the flocculated and the well-dispersed systems. Although the differences in porosity between the different layers are quite clear, they are not very great as noted earlier. It may be appropriate that conclusions regarding the structure of these layers should not consider only the porosity data. Instead, proper attention should also include the light-scattering efficiency and the gloss of the products. For example, coating layers based on color 3 had a higher porosity than those based on color 5, although not as high as layers from colors 2 and 4. The rheological properties, the light-scattering ability, and the gloss data did not indicate any greater differences between system 3 and 5, however. This may suggest that the degree of flocculation was not very pronounced in color 3.

The flocculated systems also produced coating layers on the polyester films with a greater light-scattering efficiency. In principle, this is beneficial for the optical covering ability of

the layer. The light-scattering coefficient appeared to reflect the porosity of the coating layers on the polyester film.

The preparation technique significantly affected both the rheological properties of the coating colors and the porosity and light-scattering efficiency of the coating layer. In spite of this, it was not possible to distinguish any effects on the brightness and opacity of the coated papers. The gloss was clearly higher when coating the papers with the aggregated colors, however. This may be due to the higher porosity of the corresponding coating layers. The higher porosity made them more deformable and therefore easier to calender and also thicker due to the bulkier structure of these layers. For the optical properties other than gloss, the conclusion now must be that the preparation technique only had a very slight influence, however. There may, of course, be other situations when this is not true. For instance, if the brightness of the base paper had been lower, it might have been possible to distinguish differences in optical performance. The positive effects of the changes in the structure of the coating layers caused by alterations in the preparation of the colors were obviously counteracted by or less important than the effects of other processes taking place during the coating of the paper. One such obviously important process is the surface movement or roughening of the base paper when it is contacted with the liquid phase of the coating color (23, 24). This surface movement can have a pronounced influence on the structure and covering ability of the coating layer. It is possible that this phenomenon to some extent overshadowed the effects of the preparation technique. One should also remember that the consolidation of the coating layer proceeded at a

much higher rate—partly also under the influence of a blade pressure—when coating the papers with the high-speed laboratory coater compared to the polyester films coated at room temperature. This may also influence the structure and the properties of the coating layer.

CONCLUSIONS

The manner of preparing coating colors of conventional compositions can strongly influence the rheological properties of the colors. We interpret the changes in these properties as a flocculation of the suspensions governed by the interactions between the components of the system. The flocculation increased the porosity of the dry coating layers and improved their light-scattering efficiency on a polyester film. When using these colors for coating paper with a high-speed laboratory coater, the benefits of changing the preparation procedure appeared too small to produce any significant improvements in the brightness and opacity of the coated paper. The gloss of the coated paper after calendering was higher when using aggregated coating colors for the coating, however.

TJ

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