The Effect of Yellowing Inhibitor Total Charge on The Rheology of Paper Coating

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Received: 19.4.2002, Final version: 7.10.2002

Abstract:
New compounds, called inhibitors, provide anti-yellowing effect for mechanical pulps and papers, when added to the coating formulation. The rheology of coating mixtures, which contain clay, ground calcium carbonate, starch, latex, inhibitors and other minor additives, affects to a certain extent the final quality of the coated paper. The purpose of this work is to investigate the effect of the total weight charge of inhibitors, when present between coating color ingredients, on the thixotropy and visco-elasticity of the mixture and other rheological properties. We also study the degree of interaction between inhibitor and coating ingredients using Transmission Electron Microscope (TEM) and water retention measurements.

For the industrially preferred inhibitor system of low RS/UVA ratio and high total charge the coating formulation will have a significant increase in the general value of viscosity and a clear and profound shear-thinning behavior. Under the desired conditions of higher total charge the coating formulation acquires significant thixotropic behavior. A higher level of energy is required to coat such formulation. The elastic modulus increases with frequency. This rise in the elastic modulus reveals the increasing interaction between particles in the coating formulation. The total charge does not affect the resistance of a coating color to the applied stress. The coating color with low total charge has the strongest water holding capability decreasing with increasing total charge.

Zusammenfassung:
Kürzlich eingeführte Hemmstoffe zeichnen sich durch einen Anti-Vergilbungs-Effekt für Zellstoffe und Papiere aus, wenn sie dem Papierbeschichtungsmaterial hinzugefügt werden. Die Rheologie von Beschichtungsmischungen, die Ton, Grund-Kalzium-Karbonat, Stärke, Latex, Hemmstoffe und andere Nebenbestandteile enthalten, beinflussen jedoch zu einem gewissen Grad die Qualität des fertigen beschichteten Papiers. Das Ziel der vorliegenden Arbeit besteht darin, den Einfluss des Hemmstoffanteils - sofern er in den Beschichtungsfarben vorhanden ist - auf die Thixotropie, die viskoelastischen und andere rheologische Eigenschaften der Mischung zu untersuchen. Wir studieren auch den Wechselwirkungsgrad zwischen Hemmstoff und Beschichtungsinhaltsstoffen durch den Einsatz eines Transmissionselektronenmikroskops (TEM) und anderer Verfahren.

Für das industriell bevorzugte Hemmstoffsystem mit niedrigem RS/UVA-Verhältnis und hoher Gesamtbeladung wird sich die Beschichtungsmasse durch einen signifikanten Anstieg der Viskosität, und ausgeprägtes scherverdünnendes Verhalten auszeichnen. Daher wird auch mehr Energie zum Herstellen dieser Substanz benötigt. Der elastische Modul steigt mit der Frequenz. Die Beschichtungsfarbe mit der niedrigsten Gesamtbeladung besitzt die stärkste Fähigkeit, Wasser zu speichern.

Résumé:
De nouveaux composés appelés inhibiteurs apportent un effet anti jaunissant aux papiers et pâtes mécaniques, quand ils sont ajoutés à la formulation des peintures. La rhéologie des mixtures pour revêtements qui contiennent de l'argile, du carbonate de calcium primaire, de l'amidon, du latex, des inhibiteurs et d'autres additifs mineurs, affecte jusqu'à un certain point la qualité finale du papier peint. Le but de ce travail est l'investigation de l'effet du poids total de la charge en inhibiteurs, quand ils sont présents parmi les ingrédients colorant la peinture, sur la thixotropie et la viscoélasticité du mélange, de même que sur d'autres propriétés rhéologiques. Nous étudions aussi le degré d'interaction entre l'inhibiteur et les ingrédients de la peinture en utilisant la microscopie électronique en transmission (TEM) et des mesures de traitement d'eau.

Dans le cas d'un système inhibiteur possédant un bas ratio RS/UVA et une charge totale élevée, ce qui est du point de vue industriel privilégié, la formulation du revêtement présente une augmentation significative de la valeur générale de la viscosité ainsi qu'un clair et profond comportement rhéoamincissant. Avec les conditions désirées de grandes charges, la formulation acquiert un comportement thixotropique significatif. Un plus grand niveau d'énergie est requis pour appliquer de telles peintures. Le module élastique augmente avec la fréquence. Cette augmentation révèle l'augmentation de l'interaction entre les particules de la formulation du revêtement. La charge totale n'affecte pas la résistance de la peinture à l'application d'une contrainte. La peinture possédant une charge totale faible montre la plus forte rétension d'eau qui décroît avec l'accroissement en charge totale.

Key words: inhibitor, mechanical pulp, clay, rheology, microstructure

DOI: 10.1515/arh-2002-0016

1 INTRODUCTION

The manufacture of paper from wood is a complex process, which involves a good number of stages; each is based on particular physico-chemical principles. Wood has to be turned into tiny cellulose fibers. This can be achieved either by the technology of chemical dissolution or by the technology of mechanical breakage. The resulting pulps are correspondingly called either chemical or mechanical pulps. Mechanical pulps are produced by either the grinding or the refining methods. In its original form wood is structured from cellulose fibers and other components, which are bonded by lignin. In chemical pulping lignin dissolves during the process. In mechanical pulping fragments of lignin remain in the resulting pulp. It should be noted that chemical pulps are produced at a much higher cost than mechanical pulp. Chemical pulps are used in the production of high end paper products. Mechanical pulps are used in the production of newsprint with low strength and poor brightness stability. Research, which aims at improving the potential for use of mechanical pulps, is of significant economical importance for the Pulp and Paper Industry.

After screening and bleaching processes, a wet paper sheet is produced from pulps and water in a compressor. This is followed by size pressing, where certain additives are added as a wet surface-treatment, drying and calendaring. During the initial processes water content of the paper web is gradually reduced, and raw paper is produced after a drying process. Depending on the target use of the paper product, raw paper is subjected to a coating process. Paper is treated with solutions, which provide the final product with the required characteristics of color, smoothness of surface, and brightness. These solutions are usually referred to as paper coating colors. Paper coating colors are mixtures of dispersed colloidal pigment and latex binder particles along with soluble co-binder species. These different components can associate with each other to generate different types of "wet coating structures."

The long-term preservation of paper with the same qualities of brightness and color is one of the important requirements for certain paper products. A major problem limiting the wider use of mechanical pulp is the sensitivity of lignin-containing pulps to yellowing induced by ultraviolet (UV) light. The exposure of the final paper product to the Ultra Violet wave-range of light excites the molecules of lignin, producing free radicals. These radicals oxidize with oxygen to produce yellow products. In brief, papers made from mechanical pulp turn yellow when exposed to light, creating a major barrier for using high yield pulps in papers that require long-term brightness stability [1-3].

Many methods have been explored to improve the light stability of lignin containing pulps [4]. These include chemical modification of the lignin structure [5-7], adding UV absorbers [8], adding radical scavengers, or adding a combination of UV absorbers and radical scavengers [9-11].

One of these methods is using inhibitors as part of a pigmented coating formulation. It is a novel yellowing inhibition system [14,15], which gives unprecedented light stability to lignin-containing papers. This inhibitor system contains an Ultra Violet Absorber (UVA) and a Radical Scavenger (RS). UV absorbers are normally used in different applications to stabilize paints, automotive coatings and plastics. In paper production they promise to be a yellowing inhibitor of mechanical pulp. The idea is to add them to the coating colors in the paper production machine. Figure 1 [13] shows a schematic of how the com-

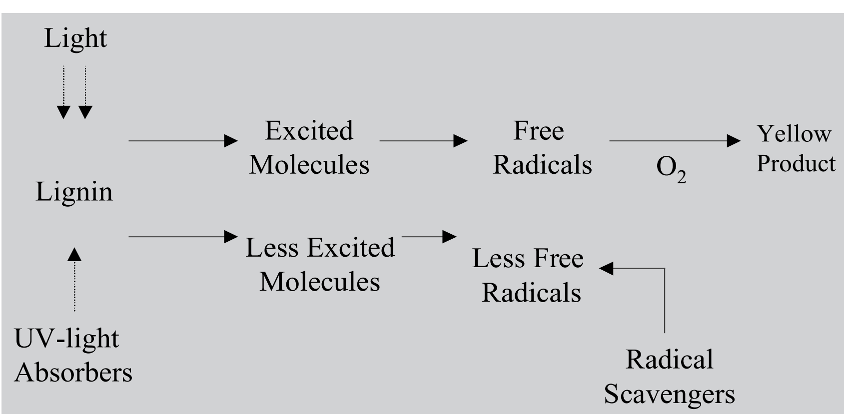


Figure 1: Mechanism of inhibition.

bination UVA and RS works. The UV absorber absorbs most of the UV light. The radical scavengers eliminate the production of most of the remaining free radicals.

The application of this inhibitor system requires the addition of two components, UVA and RS. Consequently, there are three parameters associated with this addition. One is the ratio of RS component to UVA component. The other parameter is total charge that is the total amount of the two components added to the coating formulation. The third parameter is the coat-weight or the amount of the total coating formulation per area of the paper coated. Previous studies show that these parameters have direct impact on the stability of paper brightness, in the following decreasing order: total charge > coat-weight > RS (radical scavenger)/UVA (ultra-violet absorber) ratio. Therefore the total charge has the largest effect on inhibition of paper yellowing [16].

In paper production the paper web moves with rather significant speeds of approximately 1500 to 3000 meters/min. Delivering the coating colors to the paper web while securing complete wetting under these circumstances is a challenge. Due to this extremely high inertia and the dependence of the quality of coating on the fluid mechanics of the process, this liquid coating process is dominated by the rheological properties of the coating material. Coating colors have their own set of rheological properties. Any additional additives change in one way or another these properties. To Properly understand the use of a paper yellowing inhibition system; it is important to reveal the effect of adding the inhibitors on the global properties of the resulting coating mixture. The study of the rheological characteristics of coating color suspensions has been the subject of a number of publications [17-21]. These studies have shown that most coating formulation have the same qualitative behavior with shear rates, acting initially with shear-thinning and turning at higher shear rates to shear thickening behavior.

In the present work, interest is mainly focused on the effect of the different total charges of the inhibitor on the rheology of the coating color. A comprehensive rheological investigation of coating colors could lead to very useful information and provide key parameters for relating coating problems to formulation and processing. Therefore, the objective of this work is to study the role of total charge of the inhibitor on the shear viscosity, thixotropic behavior and visco-elastic properties.

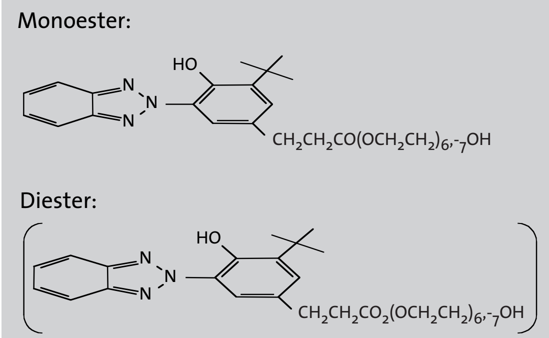


Figure 2: Structure of Tinuvin 1130™. It consistes of about 52 % monoester, 35 % diester; and was supplied as 87 % solution in polyethylene glycol (M_W = 300).

2 DESCRIPTION OF EXPERIMENTS

2.1 EXPERIMENTAL SAMPLES

The inhibitor system contained a UV absorber (UVA), and a radical scavenger (RS). A hydroxylamine salt [15] was used as a radical scavenger. An alkylene oxide substituted hydroxyphenyl benzotriazole, a commercially available UV absorber (UVA, trade name Tinuvin 1130®), was used together with the radical scavenger. The UVA consists of about 52 % monoester, 35 % diester, and was supplied as an 87 % solution in polyethylene glycol (average molecular weight of 300), as shown in Fig. 2.

The formulation, obtained from Abitibi Consolidated, contained 20 %, 80 % clay, covercarb (ground calcium carbonate, (gcc), clay, consequently, starch, latex, and some minor additives, as shown in Table I. Dry astraplate has been dispersed into cold water to obtain 70 % slurry

Table 1: Coating color components.

Chemical Name	Purpose	Solid Content [%]	Parts
Covercarb (ground calcium carbonate)	Pigment	71.6	80
Astraplate (delaminated clay, kaolinite)	Pigment	70	20
Penford Gum 280 (Starch)	Binder	20	6
Acronal S728 (Latex)	Co-binder	49.4	12
KZCote (Additives)	Cross-linking agent	100	0.5

(clay), and then added to covercarb (gcc) while mixing at high shear for 10 mins. At low-shear of mixinger, the following additives penford gum 280 (starch), acronal 728 (latex), kzcote (starch) have been added and mixed well. Then the components of the inhibitor system were added. UVA was added first, followed by RS. The pH of the final coating was adjusted to 8.7 with 50 % NaOH. The solid content was 55 %.

The range of the total concentration of yellowing inhibitors total charge was 0.2 % to 1 %. A concentration less than 0.2 % proved to have no effect on yellowing, and a concentration more than 1% is proved to have a negative effect on the coating operation, producing problems in the runnability.

2.2 RHEOLOGICAL MEASUREMENTS

All tests were conducted on the Haake rheometer RS100, which is computer-controlled. The apparatus has three modes of operation. Samples can be tested under controlled rate (CR) mode, controlled stress (CS) mode, or oscillation (OSC) mode. Software packages were used to control test routines and data analyses. All tests were conducted using a cone and plate system. The cone diameter was 35 mm while its angle was 4 °. The gap at the tip of the cone was 0.137 mm. All the measurements were conducted at a constant temperature of 25 °C. Hercules DV-10 Viscometer was used to measure the viscosity at very high shear rate.

The water retention property of coating colors was measured using a Gravimetric Water Retention Meter. A piece of pre-weighted blot paper was first placed on a rubber supporting plate. Then, a polycarbonate-based membrane with a pore size of 5 μm was placed on the top of the blot paper followed by placing a sample holder. Two mL of coating colors wereas transferred to the sample holder. A pressure of 15 psi was applied. as a function of the time. Finally, the blot paper was weighed again and the weight difference of the blot paper before and after absorbing water was taken as the water loss of the sample.

We also have applied advanced cryofixation and freeze-fracture techniques. By using cryogenic instrument we were able to freeze the coating suspensions in their original stage. The frozen samples were fracture frozen in a BAF 060 Freeze-Fracture system. Platinum carbon replicants were prepared by shadowing technique and the replicant was investigated in TEM. For rapid freezing, the ultra structure was preserved and the interaction between the actual particles could be absorbed in TEM at a resolution of 1nm, which is the magnitude higher than conventional SEM.

3 RESULTS AND DISCUSSION

3.1 RHEOLOGICAL PROPERTIES

A yellowing inhibitor system is composed of an ultra violet absorber and a radical scavenger. Two of the important factors in the application of the system are the ratio of the radical scavenger to the ultra violet absorber in the composition and the total charge of the inhibitor system within the coating formulation. In our previous work [16] we concluded that the industrially preferred parameters are a low RS to UVA ratio and a high total charge of inhibitor. Guided by these conclusions the rheological study of the effect of the increase in total charge of inhibitor should reveal the rheological advantages and disadvantages of adding the inhibitor to the coating formulation.

Figures 3 to 5 show the results of measurements of the steady shear viscosity of the coating formulation with different total charge of inhibitor. The chosen total charges were 0.2, 0.6 and 1 % within the coating formulation. These charges correspond to the practical application of the yellowing inhibitor system. Two simple conclusions were observed in Fig. 3. The general value of viscosity increased more significantly in the range of total charge 0.6 to 1. Although at lower shear rates samples with all total charges

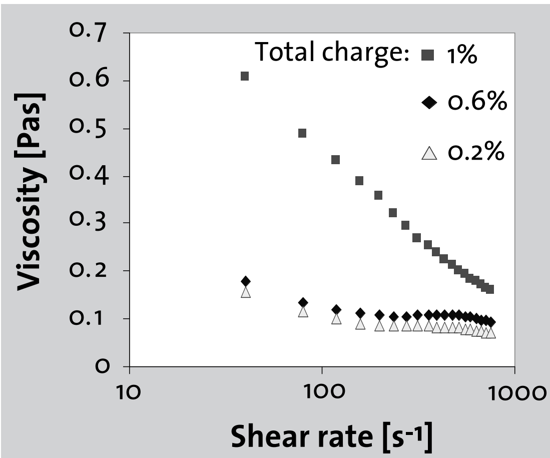
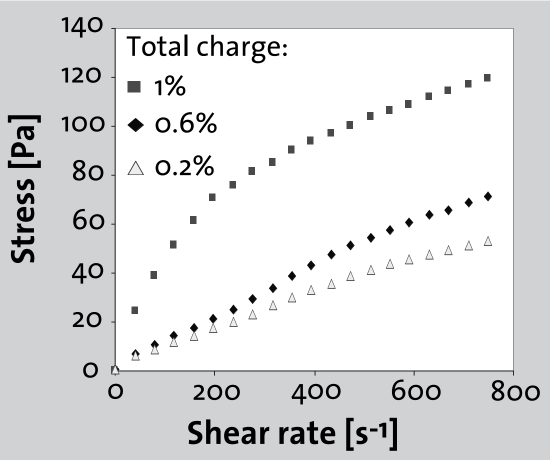
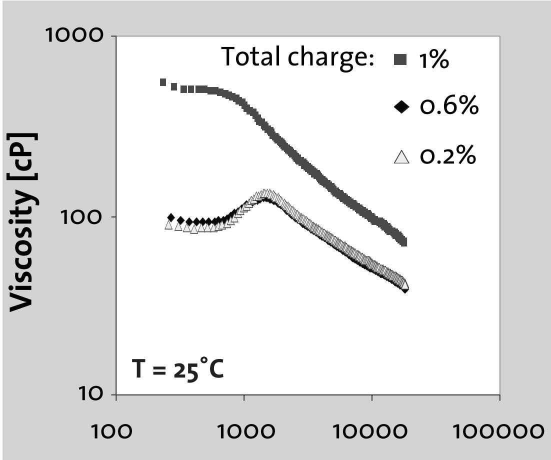


Figure 3: The viscosity as a function of shear rate for different total charges.





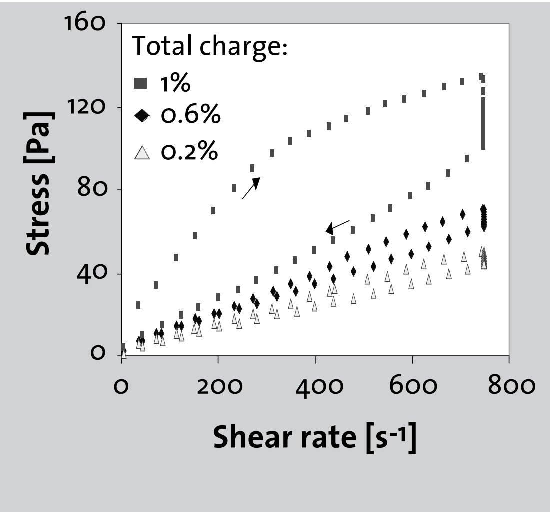


Figure 4 (left above): Stress is a function of shear rate for different total charge.

Figure 5 (right above): The viscosity as a function of high shear rate for different total charge.

Figure 6 (left below): Thixotropic behavior of coating formulation using different total charge.

exhibited decreasing viscosity with rising shear, the sample with the lower total charge 0.6 showed some noticeable increase in viscosity, that is shear-thickening, between shear rates of about 800 and 900 s^{-1}. In general, a sample with a total charge of 1, that is the highest charge, exhibited (Fig. 4) a clear and profound shear-thinning behavior over the range of shear rates up to 1000 s^{-1}. Measurements on the Hercules DV-10 viscometer covered a shear rates range of up to about 18000 s^{-1}. All samples showed a consistent (Fig. 5) decrease in viscosity with rising shear rates. We may conclude that for the industrially preferred inhibitor system of low RS/UVA ratio and high total charge the coating formulation will have a significant increase in the general value of viscosity, a clear and profound shear-thinning behavior, and a consistently decreasing viscosity for very high shear rates.

In our rheological study we conducted measurements to reveal the thixotropic behavior of the samples. Materials that exhibit thixotropic behavior have consistency, which depends on the duration of the shear applied to them, as well as on the rate of shear. The structure of the materials breaks down under shear, but builds up again with the gradual removal of shear. A state of dynamic equilibrium may be reached when the build up of structure equals the rate of breakdown [12]. Thixotropic measurements were conducted under the controlled rate CR mode of the Haake rheometer. The stresses and shear rates were recorded. The resulting up-curve was obtained in the process of gradually increasing the shear rate. After reaching the assigned maximum rate, a gradual decrease gives the down-curve, which should coincide with the up-curve in the case of time independent rheological behavior. For thixotropic solutions the down-curve is different from the up-curve. In this case the two curves form a "hysteresis" area A, which is a quantitative measure of the degree of thixotropy. The hysteresis area has the dimensions of energy over volume, that is the energy required to break down the thixotropic structure of the solution.

Figure 6 displays the hysteresis areas obtained for the different tested samples. It is evident that solutions containing lower total charge of the inhibitor system 0.2 % and 0.6 % have noticeable but rather small hysteresis areas. However, the higher total charge of 1 % introduces a significant thixotropic effect, manifested by a sizable hysteresis area. We conclude that under the desired conditions of higher total charge the coating formulation acquires significant thixotropic behavior. A higher level of energy is required to coat such formulation. This may be a positive feature in coating at lower speeds, such as in paints. However, in high-speed paper coating processes the need to affect a reasonable duration of shear to break down the thixotropic structure may indeed impede the complete wetting of the substrate, and could predictably lead to coating defects.

The creep and recovery measurements introduce the response time to the stress dependency of both the viscous and the elastic behavior of solids and fluids. Constant shear stress of values 1 and 0.5 Pa was applied each for 150 s to the coating color. Following this period of time, the applied stress was brought instantaneously

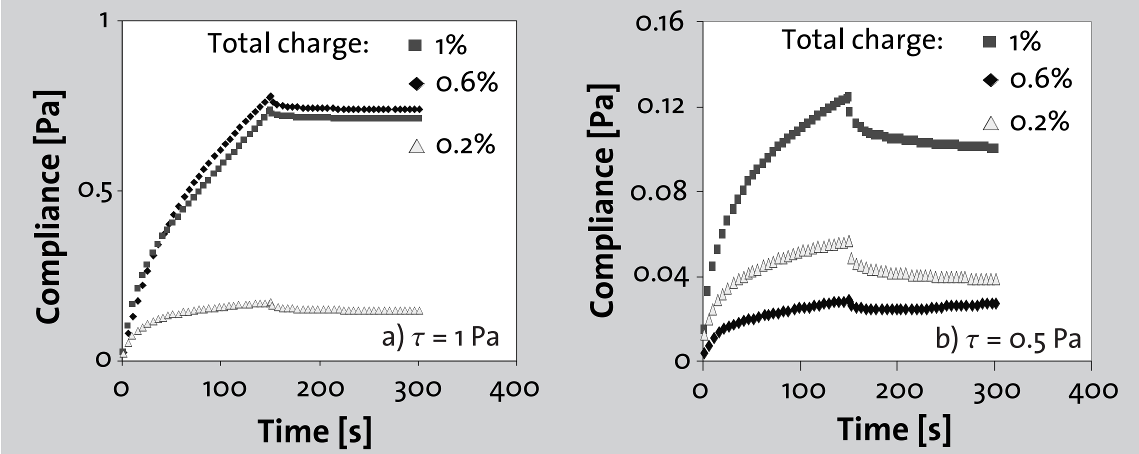


Figure 7 (above): Creep curves for coating formulation of different total charge.

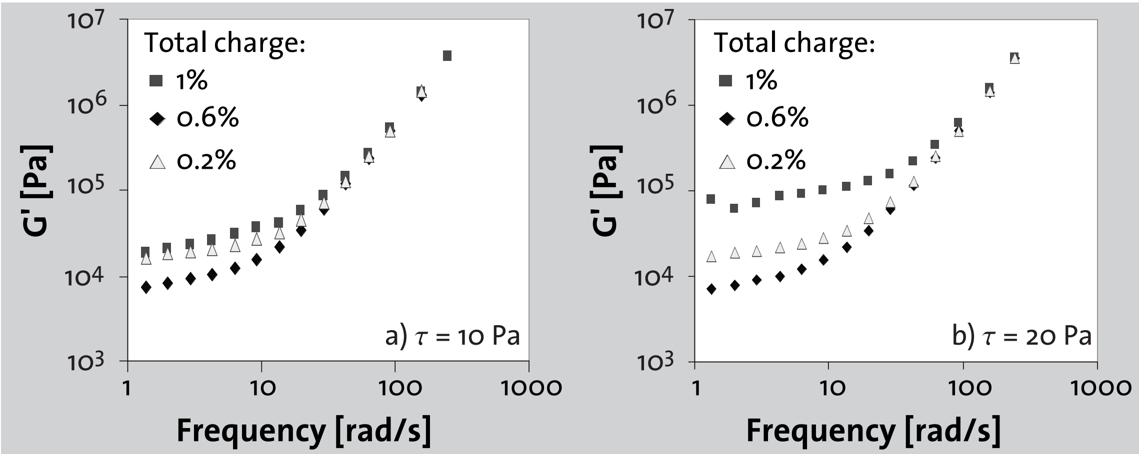


Figure 8 (middle): Elastic modulus curves for coating formulation of different total charge.

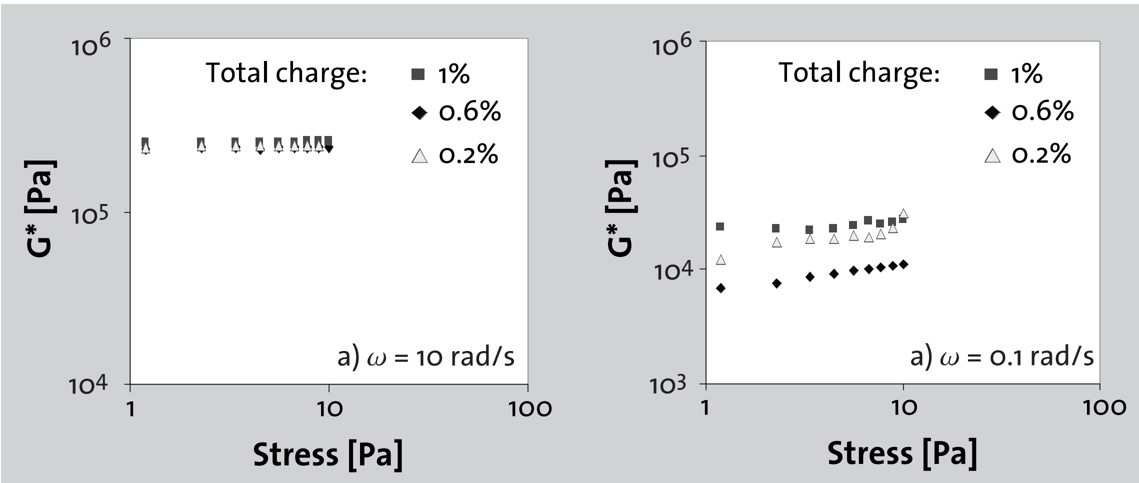
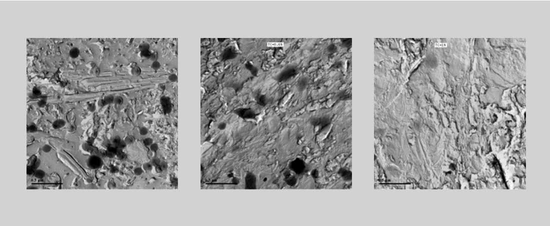


Figure 9 (below): Viscoelasticity of coating formulation with different total charge.

to zero. The response of deformations was recorded over a total period of 300 s. The stress in the recovery phase of this test must be set to zero for full recovery to reach a final permanent strain level. Figure 7a shows that the creep deformation for total charges of 1, 0.6 and 0.2 %. It shows that the deformation for total charge of 1 and 0.6 is approximately the same, and is significantly higher than for 0.2 %. When the stress was reduced to 0.5 Pa (Fig. 7b) the creep deformation dramatically decreased although it remained relatively higher for total charge of 1 %. It is also noted that the deformation for 0.6 % total charge became less than the deformation for 0.2 %.

The variations of the elastic moduli with frequency for different total weight inhibitors are presented in Fig. 8. The measurements were carried out at stress 10 and 20 Pa. The elastic modulus increases with frequency. This rise in the elastic modulus reveals the interaction between the particles in the coating formulation. Overall the elastic modulus of a coating color with a total charge of 1 % is markedly larger than those for colors with total charges of 0.2 and 0.6 %. On the other hand increasing the stress to 20 Pa gives further increase in the elastic modulus of the



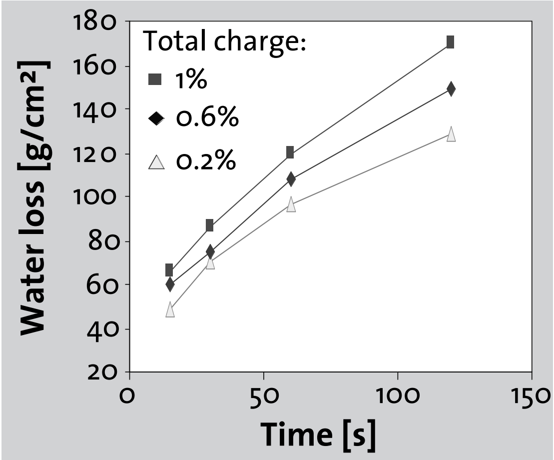


Figure 10 (right): Water loss of coating formulation with different total charge.

Figure 11 (left): Coating formulation including different total carge: a) 0.2%, b) 0.6%, c) 1%.

color with 1%, indicating an increase in the interactions between particles. The frequency tests also reveal the coating formulations exhibit yield stresses.

Figure 9a displays the linear visco-elasticity of colors with all tested total charges at frequency 10 Hz. It is also noted that the total charge does not affect the resistance of a coating color to the applied stress. However at low frequency of 0.1 Hz (Fig. 9b) the resistance of coating colors is dependent on the amount of the total charge. The resistance of the coating colors increases with increasing the applied stress. While the linear visco-elasticity indicates that the coating color is deformed without destroying the bonds of the molecules, at low frequency, the nonlinear visco-elasticity reveals that the coating color deformation is accompanied by the destruction of bonds between particles due to the applied stress.

3.2 TEM MICROGRAPHS

Comparison of the TEM micrographs of the coating formulations containing 1, 0.6 and 0.2 % total charge are shown in Fig. 11. They indicate that the interaction between 0.2 % total charge of inhibitor and coating color is different from that for colors with 0.6 % and 1 % total charge. This is apparent from the microstructure. The micrograph (Fig. 11b, c) appears more densely packed in comparison to that for coating color with 0.2 % total charge. Coagulation occurs from the bridging between the inhibitor and coating formulation particles. According to this micrograph, it is clear that the concentration of inhibitor is predominant to the aggregation effect. Therefore, high magnification transmission electron microscopic (TEM) pictures were used as a qualitative indication of the extent of interparticle structuring and compare it to the rheological measurement.

3.3 WATER RETENTION - EFFECT OF INHIBITOR

Figure 10 shows measurements taken at various time and total charge of inhibitor. The same pressure was used for all experiments. Loss of water between application and metering will lead to a high solids level. The coating formulation including 1 % total charge has the highest water loss. This indicates that the packing of the coating color particles becomes more open at high levels of total charge addition. Because of the more open structure, the water drains faster causing less water retention and increased viscosity. Addition of 0.6 % total charge of inhibitor to the coating formulation resulted in the particles becoming more densely packed than in the case of 0.2% total charge. The coating color with total charge (0.2 %) has the strongest water holding capability followed by that with 0.6 % and finally with 1 %.

4 CONCLUSIONS

We may conclude that for the industrially preferred inhibitor system of low RS/UVA ratio and high total charge, the coating formulation will have a significant increase in the general value of viscosity and a clear and profound shear-thinning behavior. We also conclude that under the desired conditions of higher total charge the coating formulation acquires significant thixotropic behavior. A higher level of energy is required to coat such formulation. The elastic modulus increases with frequency. This rise in the elastic modulus reveals the increasing interaction between particles in the coating formulation. It is also noted that the total charge does not affect the resistance of a coating color to the applied stress. The coating color with low total charge has the strongest water holding capability decreasing with increasing total charge.

ACKNOWLEDGMENT

The authors acknowledge the valuable discussions with Dr. Zhirun Yuan, and permission to use equipment in PAPRICAN laboratories.

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