

Depletion of coating color components in the blade coating process circulation

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ABSTRACT: Coating color stability, as defined by changes in its solid particle fraction, is important for runnability, quality, and costs of a paper coating operation. This study sought to determine whether the size or density of particles is important in size segregation in a pigment coating process. We used a laboratory coater to study changes in coating color composition during coating operations. The results suggest that size segregation occurs for high and low density particles. Regardless of the particle density, the fine particle size fraction ($<0.2\ \mu\text{m}$) was the most prone for depletion, causing an increase in the average size of the particles. Strong interactions between the fine particles and other components also were associated with a low depletion tendency of fine particles. A stable process and improved efficiency of fine particles and binders can be achieved by controlling the depletion of fine particles.

Application: By controlling size segregation and depletion of fine particles and binder during paper coating operations, mills can achieve a more stable process and product quality, improved binder efficiency, and cost savings.

In paper coating, a dispersion of inorganic and organic solid particles in a thickened water medium is applied on paper to improve smoothness and printing properties. A coating color is applied on to a running paper web with an application unit; coat weight (film thickness) is adjusted with a metering element; and surplus coating color, typically 90%-95%, is recirculated, forming a recycling stream.

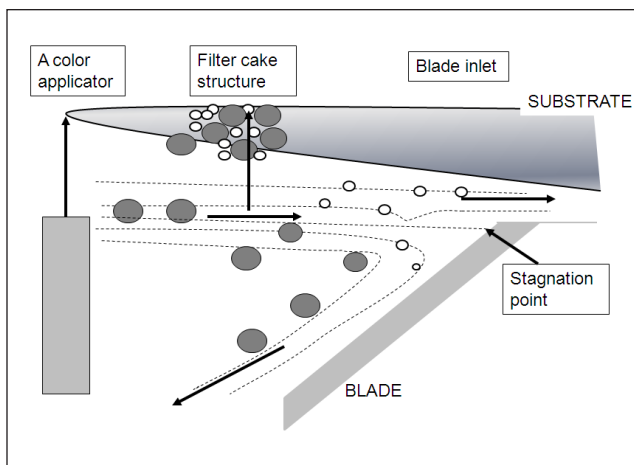
The term “segregation” is used to describe the solid phase separation into fractions that vary in characteristics, such as particle size, shape, or density. In the paper coating process, the dry solids content of a coating color increases with recirculation over time, and the solid particles’ composition also changes. Coating color dewatering has been studied widely, but segregation is not a well-recognized phenomenon. Typically, average particle size increases as a result of depletion or forming clusters of latex binder (colloidal) particles and the fine fraction of the pigment particles. This phenomenon is defined here as particle size segregation. Changes in a particle size distribution affect the coating process runnability and stability. If size segregation and depletion of fine particles, such as binder particles, can be controlled, a more stable process, improved efficiency of binder, and stable quality can be achieved, along with cost savings.

For changes such as particle fraction movement to occur, driving forces for material movement must exist, as must coating color properties that allow particle fractions to move differently in relation to each other [1].

Various flow fields and pressure pulses in a blade coating process can cause particle segregation. Pressure penetration during application and metering, as well as capillary penetration caused by substrate absorption, yield the primary driving forces to particle movement toward the substrate, perpendicularly to the web running line (z-direction). When a fluid is

dragged over a surface, the pattern of the flow will vary with the velocity, the physical properties of the fluid, and the geometry of the surface (flow boundaries) [2]. Particle migration from more advantageous energy distribution in a stress field also might take place and cause different particle distribution in the z-direction [3]. Small particles get closer to the boundaries than do large particles, and thus small particles are concentrated near boundaries. The small particles can push the large particles outward, and the large particles would be selectively metered away from the web [3-6]. The gravitational force is too slow to generate size segregation of particles in a high-speed coating operation [7,8]. The diffusive forces are larger than the gravitational force [9]; they make small particles, such as binders (100-300 nm) and thickeners (5-10 nm), which are more mobile relative to large particles [9-12]. If the direction of flow changes rapidly, or the suspension is accelerated or decelerated at high shear rates, the greater inertia of large solid particles forces the particles to cross the fluid streamlines or to lag behind the fluid flow. Gagnon et al. [8] proposed pulse dewatering as a particle segregation mechanism. As large particles get left behind because of their inertia, they are separated from the liquid phase and from small particles near the stagnation streamline because of the pulse dewatering.

A difference in particle size between components increases segregation tendency [13]. Kline [1] and Al-Turaif and Bousfield [11] found that latex binder movement toward the base paper was greater in coatings with narrow particle size distribution pigments than in those with broad ones. Particle flocculation or agglomeration decreases size differences between the components, because flocs or aggregates are the basic structural units [9]. The structures and agglomerates can be formed by electrolytes [1,14], by destabilizing agents [15], and in other ways.



1. The areas for separating particles in a coating process. The arrows show direction of particle movement.

The structure of a coating color also might be affected by shearing it in narrow gaps, causing shear-induced aggregation of particles [14,16]. Brownian motion can promote the formation of flocs [14]. Dissolved polymers, such as thickening agents, have been found to prevent latex particle mobility [12,17-20]. Two mechanisms have been proposed: 1) thickeners flocculate the particles [10,21-23] or 2) thickeners form a network structure that efficiently reduces latex mobility [24,25]. Water functions as a carrier for the solid material and particles smaller than 1 micron move with the fluid phase [8,26]. A filter cake forms on the paper surface as a result of dewatering. Gagnon et al. [8] found that the degree of filter cake formation would not influence particle segregation based on the inertia of particles, but it manifests a limit to the magnitude of dewatering. Low solids content, low viscosity, and high application volume should allow more opportunity for particle movement before immobilization.

Figure 1 illustrates proposed mechanisms of particle size segregation in the literature previously discussed. The coating processes comprise application and metering units. Speed and type of application unit determine the delay time between the units. In the application, a part of the coating color penetrates into base paper and coating color starts to dewater. A filter cake on the paper web surface forms as a result of dewatering.

However, simple filter cake formation does not explain size segregation, because all particles in the filter cake get carried out under the blade. Gagnon et al. [8] attributed size segregation in blade coating near the stagnation point to inertia: large particles, because of their large inertia, get left behind; small particles and large particles are separated from the liquid phase, and large particles are separated from small particles by pulse dewatering.

The motion of particles with different characteristics in a flow can be evaluated by considering the particle Reynolds number, Re_p , for an individual particle. The Re_p signifies the ratio of inertia effects to viscous effects influencing the particle in a flow [2]:

$$Re_p = \frac{\rho v d}{\eta} \quad (1)$$

where

Re_p = Reynolds number for a particle, -

ρ = particle density, kg/m³

v = velocity difference between a particle and fluid surrounding it, m/s

d = particle diameter, m

η = liquid phase viscosity, mPas

The inertia forces become more important when Re_p increases (i.e., the particle density increases, the velocity difference between a particle and fluid surrounding it increases, and the particle size increases). Properties of individual components determine their response in the flow and the tendency of particles to segregate during a coating process.

Furthermore, the size segregation can be enhanced by the following mechanisms:

- On the surface of the filter cake, small particles can push larger particles away [3-4], creating a fine material gradient in the nonimmobilized coating color, thus increasing size segregation.
- The blade geometry and the gap between filter cake and blade can limit the size of particles fitting in the gap, but only if the filter cake thickness is large in comparison with the blade to base paper gap.

These mechanisms influence the coating color repeatedly over time, as the coating color recirculates in the application unit. The number of recirculations is determined by the volume of coating color in the applicator recirculation, as well as the applied and metered coating amounts. Common coating heads have recirculation rates of 30:1 [27]. The higher the machine speed, the larger the dewatering pulse, the inertial force, and the size segregation.

In this study, we sought to determine whether the size and density of particles are important in size segregation in the paper coating process.

MATERIALS AND METHODS

The coating colors used for this study consisted of a coarse calcium carbonate pigment (HC-60, 60% <2 μ m, density 2710 kg/m³) from Omya (Oftringen, Switzerland); a fine pigment, either a high-density mineral pigment (Setacarb HG, mean size 0.45 μ m, density 2710 kg/m³, Omya) or a low-density plastic pigment (DPP 3740, mean size 0.45 μ m, density 1050 kg/m³) from Dow Chemical (Midland, MI, USA); and a styrene-butadiene latex binder (Dow 920, particle size 0.13 μ m). The thickener we used was carboxymethyl cellulose (FF-10, Mw 80 kD) from CP Kelco (Atlanta, GA, USA); which thickens in the aqueous phase and is not expected to adsorb onto pigment and latex particles [28,29]. We also used a tetrasulphonated optical brightener, Blankophor P, (Blankophor; Leverkusen, Germany).

Grammage, g/m ²	80
Roughness, Bendtsen, mL/min	200
Thickness, μ m	100
Air permeability, Bendtsen, mL/min	640

I. Base paper properties.

COMPONENTS, pph	High Density Color	Low Density Color
Coarse pigment	80	91
Fine, high density pigment	20	
Fine, low density pigment		9
Thickener	0.8	1.4
Latex	10	10
Optical brightener	1	1

II. Coating color formulations.

The coatings were applied to unsized, uncoated fine base paper. **Table I** shows the characteristic properties of the base paper.

Table II presents the coating color formulations. The dosage of the fine pigment fraction was kept constant at 20% by volume. The coating colors were adjusted to the solids volume concentration of 40%. The amount of thickener was adjusted according to the low shear viscosity, measured at 100 rpm, to a level 800-900 mPas (25°C). The pH of the coating colors was adjusted to 8.5 by sodium hydroxide.

The coating trials were carried out with a laboratory-scale, continuous roll-to-roll coater. The speed of the coater was 70 m/min. The coating was applied on the web with a roll applicator and the coat weight adjusted by a stiff blade. The coated side of the web was dried with infrared and air dryers. The coating method was used to evaluate water retention and the particle segregation tendency by measuring the solids content and particle size distributions from the recirculated coating color. The experiment was started with a constant amount of coating color; no additional fresh coating color was added during the experimental trial run. The blade pressure was adjusted during the trial to keep a constant coat weight of 9 g/m², corresponding to 5% of the applied coating color following the paper web, and the rest being metered and recirculated back. **Table III** lists additional parameters. The increase in the solids content of the return coating color was monitored every minute, up to 4 min. The particle size distributions of the coating samples before and after the trials were characterized. McKenzie et al. [30] have presented a more detailed description of the method.

The freshly prepared coating colors were exposed to shear forces in the capillary viscometer to investigate whether the

	Angle of Blade Support, °	Blade Pressure, kPa	Coat Weight, g/m ² Average $\pm$ Standard Deviation
High density color	20	100-220	9.3 $\pm$ 1.1
High density color	20	50-220	8.2 $\pm$ 0.5

III. The coating parameters: steel blade (0.381 mm * 100 mm) and constant gap of 0.3 mm between application and backing roll.

Size Fraction, %	0 ... 0.2 μ m	0.2 ... 2 μ m	2 ... 10 μ m
Coarse pigment	5.0	45.4	49.6
Fine, high density pigment	17.3	82.4	0.3
Fine, low density pigment	0	100	0
Latex	100	0	0

IV. The share of different size fractions for the raw materials as volume percentages.

coating colors were sensitive to shear-induced aggregation and whether that is one mechanism for the particle segregation over time.

The coating colors were analyzed with several tools to further understand the segregation mechanisms. All measurements for the coating colors were performed after typical handling at constant temperature (25°C). Particle size distributions, solids content, and inorganic/organic ratio, expressed as the ash content, of coating colors were measured.

The particle size distributions of raw materials and coating colors were measured with a laser diffraction instrument. The distributions were evaluated by dividing them into the three fractions (described in **Table IV**): fine (latex + fine fraction of calcium carbonates; 0-0.2 μ m), medium (plastic pigment and mostly fine calcium carbonate pigment; 0.2-2 μ m) and coarse fraction (mainly coarse calcium carbonate HC-60; >2 μ m).

Solids content was measured with a halogen moisture analyzer (Mettler Toledo; Greifensee, Switzerland). Viscosities of coating colors were measured with Brookfield a rotational viscometer (100 rpm) from Brookfield (Middleboro, MA, USA) and with a capillary viscometer (capillary size: 50 mm / 0.5 mm) from DT Paper Science (Turku, Finland). Apparent slip velocity was calculated from capillary viscosity measurements using two capillary sizes (0.52/100 mm and 0.7/100 mm) according to the method reported by Kokko [3]. Static water retention (ÅA-GWR) was measured using a con-

stant coating color volume of 10 mL (pore size 5 mm of membrane, 30 kPa pressure, 2 min).

Immobilization solids content and immobilization time of make-down coating color were measured with a ceramic plate technique (20 mm wet coating layer) [31].

Aqueous phases of coating colors were separated by centrifuging the coating color at 3400 rpm for 4 h, after which the supernatant was decanted. The aqueous phase solids content was measured with a halogen moisture analyzer and viscosity was measured with Brookfield viscometer equipped with small sample adapter (60 rpm, spindle 18).

The stress measurement for the coating colors was done with the Eklund capillary viscometer, where the coating color was run through three different sizes of capillaries (0.52/50 mm, 0.52/100 mm, and 0.7/100 mm) and a pressure range from 1000 kPa to 17000 kPa was used.

Elastic modulus, G' , critical strain and cohesion energy for a makedown coating color were measured. Oscillation tests were carried out with a rheometer with CC27 geometry (Anton Paar; Graz, Austria). In the amplitude tests, a frequency of 1 Hz was used to characterize critical strain. We allowed a 3 min rest time before measurements were taken. The formula for the cohesion energy is given as Eq. (2) [32]:

$$\text{Cohesion energy} = 0.5 \gamma_{\text{CR}}^2 G'_{\text{LVE}} \quad (2)$$

where

Cohesion energy, mJ/m^3

γ_{CR} = a critical strain, -

G'_{LVE} = an elastic modulus in linear viscoelastic area LVE, Pa

The inorganic/organic ratio expressed as the ash content was determined by burning the coating color at 425°C for 4 h and measuring the weight loss.

RESULTS AND DISCUSSION

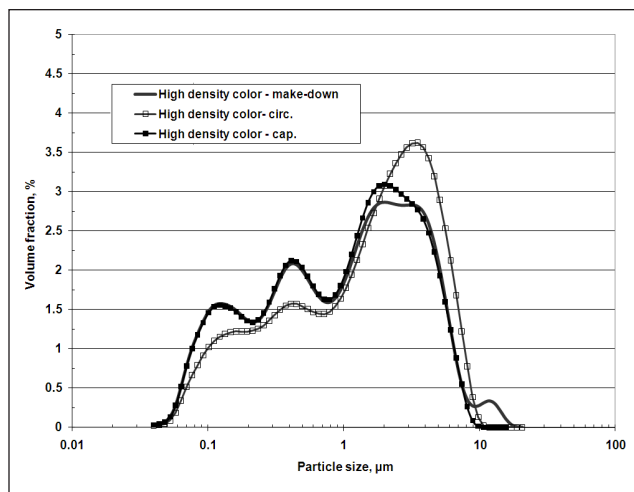
Table V presents characteristics of the coating colors. **Figures 2-3** and **Table VI** summarize the results for the changes in the particle size distributions of the make-down coating colors when sampled from the circulation flow of the laboratory-scale coating process and after exposure to the high shear stresses in the capillary viscometer.

While the solids volume fraction and low shear viscosity were kept constant, the pigment change induced changes in the wet structure of the coating colors. Results in Table V show differences in aqueous phase properties and structure parameters, such as storage modulus and cohesion energy, describing the number and cohesiveness of the structures. Table V shows that there were no significant differences in the dewatering properties between the coating colors. The immobilization properties of coating colors were similar.

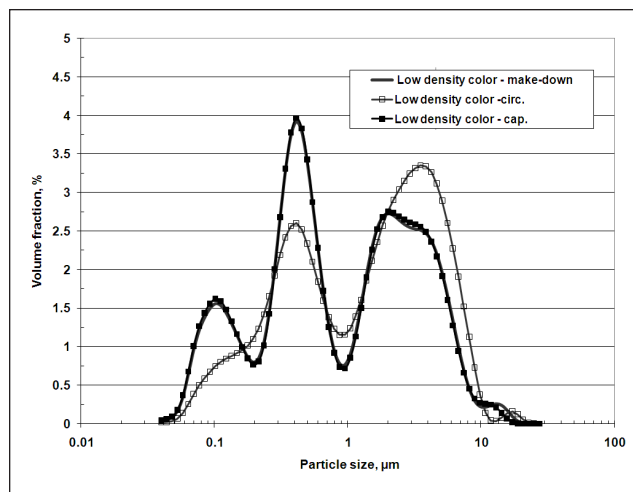
Because the thickener dosage differed between coating colors to reach constant viscosity at low shear rate, despite maintaining a constant solid volume fraction, interactions between the thickener and the used pigment systems might have differed. The surface chemistries of pigments were not characterized in this study. The dosage differences can be used to explain differences in the aqueous phase viscosities measured at low shear rate and aqueous solids contents. The apparent slip velocity also was low for the coating color with low density pigment, indicating high aqueous phase viscosity at high shear rates. Characterization of the aqueous phase is complicated by the many separa-

	High Density Color	Low Density Color
Solids fraction volume, -	0.40	0.40
Solids content, %	61.2	58.7
Capillary viscosity (500000 1/s), mPas	25	26
Water retention, ÅA-GWR, g/m ²	175	171
Water retention, Novicoater, delta-%	6.9	6.9
Immobilization time, s	8	9
Immobilization solids content, %	84.8	83.8
Immobilization solids fraction volume, -	0.70	0.71
Aqueous phase viscosity, mPas	6.9	10.7
Solids content of aqueous phase, %	2.3	3
Critical strain γ , -	0.000623	0.000432
Storage modulus G' , Pa	1190	660
Cohesion energy, mJm^3	0.23	0.06
Ash content of color before coating, %	90.6	82.4
Ash content of color after coating, delta-%	1.2	3.4
Slip velocity (at 15000 N.m ²), m/s	27	18

V. Coating color analysis results.



2. Particle size distributions of coating colors containing high density mineral pigment from make-down, after the coating process exposure (circ.) and after the capillary viscometer exposure (cap.).



3. Particle size distributions of coating colors containing low density plastic pigment from make-down, after the coating process exposure (circ.) and after the capillary viscometer exposure (cap.).

tion parameters involved, such as centrifugal forces and duration time or pressure in filtration, which might affect the composition of the separated aqueous phase.

The oscillation measurement results in Table V show that the storage modulus G' and critical strain of the coating colors containing low-density plastic pigment were smaller than those of the coating colors containing high-density mineral pigment. Although CMC has not been found to adsorb onto the calcium carbonate pigment or latex particles [28,29], the structures might originate from flocculation by depletion mechanisms.

Changes in particle size distributions of the coating colors in circulation during the laboratory-scale coating process (Figs. 2 and 3) typically show a relative decrease in the fine and medium size fractions and relative increase in the coarse size fractions. The changes were larger for low-density plastic pigment-containing formulations than for the high-density mineral pigment. Changes in particle size distributions of the coating colors after the shear stress exposure in the capillary viscometer were smaller than after the coating process exposure.

Because quantitative changes in particle size distributions were difficult to accurately evaluate from the figures, the relative changes of each fraction were calculated, as already described. Table VI shows the results.

Table VI shows that while during the coating color circulation proportions of both the fine and medium size fractions decreased, the change in the fine particle fraction was larger. The large depletion of the fine size particle fraction is in agreement with the studies by Gagnon et al. [8] and Sand et al. [10]. When comparing the coating colors, it is apparent that the fine size fraction of low density coating color changed more than the corresponding fraction of the high density coating color. No such difference between the coating colors was observed for the medium size fractions.

Change, %	Fine, %	Medium, %	Coarse, %
High density color – circ.	-24.4	-11.7	27.5
Low density color – circ.	-33.9	-12.1	34.2
High density color – cap.	0.3	-1.4	1.8
Low density color – cap.	2.5	-0.7	-0.2

VI. Relative changes of particle size fractions in the coating process (circ.) and after high shear exposure with capillary viscometer (cap.).

The coating colors containing low density pigment had higher aqueous phase viscosity, but it changed more than for the coating colors containing high density pigment. High aqueous phase viscosity has been proposed to slow the depletion of fine and light particles [1,19]. Kline [1] also found that high aqueous phase viscosity delays immobilization of a coating color, thus allowing more time for the movement. The results here support the latter; filter cake formation also can be delayed in case of high aqueous phase viscosity. Furthermore, the oscillation result suggests that there were fewer structures [23] and the structures were weaker [32] in the coating colors containing plastic pigment, thus allowing particles to move more freely.

The changes that took place during high shear exposure were small compared to the changes during coating process exposure (Figs. 2 and 3 and Table VI). We saw no clear trends among colors, and propose two explanations for this. First, there was no flocculation or aggregation of particles in the coating color, or shear-induced aggregation needed longer exposures and higher shear forces to occur. Second, if fines

were flocculated, they did not irreversibly aggregate. The flocs broke down easily under shear forces or dilution for the particle size distribution measurement destroyed the flocs.

These results support the hypothesis that the depletion of fine material is the cause for changes in the particle size distributions rather than formation of aggregates or flocs in the flow. The hypothesis is also supported by the observed changes in the inorganic/organic material ratios. The ratio increased 1.2%-3.4% units during the coating color recirculation with both coating colors, especially in the case of the coating color with low density plastic pigment.

The importance of particle inertia during the repeated pulse dewatering in a circulation process, as proposed by Gagnon et al. [8], can be estimated by comparing particle Reynolds numbers, Re_p , for coating color components. The Re_p ratios for typical particles in the different size fractions are calculated below, based on the particle densities and typical sizes noted above, and the measured liquid phase viscosities given in Table V:

- Re_p (high density pigment, medium size fraction) / Re_p (low density pigment, medium size fraction) = 4.0
- Re_p (high density pigment, medium size fraction) / Re_p (latex) = 8.9
- Re_p (high density pigment, medium fraction) / Re_p (fine fraction of high density pigment) = 4.5
- Re_p (low density pigment, medium size fraction) / Re_p (latex) = 3.5

Although the Re_p of the high and low density pigments in the medium size fraction differed by a ratio of 4.0, the difference was either not high enough to induce size segregation based on inertial effects, an unknown additional mechanism counteracted the changes, or the mechanism is not relevant for the process under consideration. The largest difference in the particle Reynolds numbers was seen between the medium size fraction high density pigment and the latex particles. However, this difference did not lead to the largest changes in the particle size distributions during the circulation experiments. This suggests that particle size is more important for depletion of particles than density. Strong interactions between the fine particles and other components also were associated with a low depletion tendency of fine particles.

Industrial formulations are hard to study because changing one parameter causes changes in many other parameters simultaneously, and therefore, the root cause of observed results is uncertain. This study was carried out with a slow speed, laboratory-scale coater. The delay times are longer and web and coating colors are colder than in full-scale coating production. However, significant changes in the particle size distributions were observed despite the short runtimes. There was a long delay between the application and metering blade, giving more time for a structure buildup than in full-scale production. At higher speeds, shear forces increase and strength of wet coating structures becomes more important. At higher

shear forces the shear-induced aggregation also could be of importance for particle size segregation.

CONCLUSIONS

Results of our study indicate that size segregation occurs for high and low density particles. Regardless of particle density in the composition, the fine particle size fraction (<0.2 μm) was the most prone for depletion, causing an increase in average particle size.

High aqueous phase viscosity itself is not enough to resist depletion. On the contrary, it can keep the color mobile longer, slowing down filter cake formation, and thus increasing depletion of fine particles. The size segregation can be decreased by controlling wet coating color properties such as interactions between components and their strength. If fine material is part of the strong structure, the depletion of fine material can be decreased. In this case, the structural unit is a cluster of particles and not individual particles.

Comparison between the coating process and high shear stress exposure led to the conclusion that, in the low-speed coating process exposure, the shear-induced aggregation was not a mechanism for particle depletion. **TJ**

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The coating topic we chose to research is not well recognized and understood, and it does not become apparent in typical laboratory- or pilot-scale studies. However, it has significant effect on stability and efficiency in production scale coating.

Our study discusses the mechanisms and effects of coating color characteristics on size segregation in pigment coating, which have not been published earlier. This is difficult to study by using industrial formulations because changing one parameter causes changes in many other parameters simultaneously. Therefore, the root cause of observed results can be uncertain. Despite uncertainties in the explanations, the depletion of fine material was significant.

The most interesting result was that both low and high density particles were depleted. We have carried out further studies about coating color characteristics and studies at high speed.

The results discussed in this paper can help researchers and mill workers understand why coating colors can have different properties in the production circulation loop than freshly prepared coating colors.



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