Peptization Control of Composite Materials Containing Water Glass for Spray Drying of Catalysts

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1 Introduction

Silica-based composite particles are complex catalyst materials that are widely used in petrochemistry. The preparation of particles that are well defined in form, composition, and chemical and rheological behavior by spray drying of suspensions remains an important industrial challenge. Here, zeolite HY, kaolin, and the cheap binder water glass are used. Experimental and model regimes of slurry peptization are analyzed based on their pH value, zeta potential, and mixing speed to control the process. The results show a pseudoplastic behavior of the silica slurries for zeta potential below 20 mV, which is explained by surface charge, hydroxyls, and pH-dependent coagulation. Thus, a quick acidification of the slurry, additional mechanical shear, and subsequent pH control during spray drying enable control of peptization kinetics.

Keywords: Peptization control, Silica-based composite particles, Slurry peptization, Spray drying, Zeolites

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The formation of stable silica-based particles is a major challenge and well-established technology in industrial catalyst manufacture, e.g., in fluid catalytic cracking (FCC). From silica/alumina catalysts in the 1940s by Houdry to zeolite-based catalysts by Union Carbide in 1959 and the stepwise development of additives and promoters until today, there is a strong economically forced development to increase flexibility in fine-tuning for industrial application [1]. Nowadays, there is a switch in focus among the research community not only towards improving catalyst performances, but also revealing their fundamental reasons [2]. Thermal and mechanical stability as well as tunable shape and porosity are important particle parameters to control fluid dynamics, energy and mass transport as well as the chemical reaction rate in oil-refining processes.

Catalyst shaping as mentioned by Schüth and Hesse includes different methods (e.g., pelleting, granulation, extrusion, spray crystallization), with one major challenge being the control of a uniform geometry and size of the final catalyst body [3]. On the one hand, the spray drying and crystallization process is strongly coupled with the fluid behavior of the spray feed, i.e., slurry viscosity [4]. On the other hand, the properties of the final product are related with the physical and chemical processes within the spray drying procedure [5]. A special focus is placed on particle size, attrition resistance, fluidizability, and thermal behavior by stable chemical bonding [2]. In this context, colloidal silica is often used as a binder in the formation of FCC catalysts [6]. This material produces chains of silicate to bind particles through the formation of stable polysilicates on their surface [7]. This gelation is observed macroscopically through the modifiable rheological behavior of the slurry, which depends on concentration, the presence of gelling agents, etc. [7, 8]. Thus, the chemical behavior of silica slurries is key to expanding knowledge of peptization and increasing control in the catalyst preparation [9].

The colloid chemistry of silicates in sol-gel processes has been studied for several decades [10]. However, the explicit fine-tuning and understanding of the infinite interactions of these binders with many other components in a catalyst grain and correlations to the preparation procedure and final grain architecture remain a focus of current research efforts [5, 11]. To the authors’ knowledge, there is no interdisciplinary approach to understand the interdepending surface chemistry of catalyst particle binding by silica coagulation considering rheological phenomena to control and fine-tune the binder interaction with zeolite Y.

Therefore, the main aims of the present study involve analyzing the surface (electro-) chemistry during agglomeration of silica- and zeolite Y, explained for multicomponent particles. In addition, the fundamental forces for particle coagulation are
described through a model that considers the particles’ surface phenomena. The resulting model is adapted to explain the influence of mechanical forces in the peptization behavior of the system. Finally, the control of particle size distribution of catalysts including water glass binder can be established through adjusting the mechanical shear, zeta potential, and pH value in a certain preparation sequence.

2 Materials and Methods

2.1 Raw Materials

The materials utilized for the formulation and characterization are commercial sodium metasilicate (hereinafter referred to as water glass) by Merck (27 wt % SiO2: 8 wt % Na2O), zeolite CVB400 HY by Zeolyst (SiO2/Al2O3 = 5.1), and kaolin by Sigma Aldrich. Hydrochloric acid (VWR Chemicals) as well as sodium hydroxide (Sigma Aldrich) were used to control the pH value.

2.2 Preparation of Slurries and Spray Drying Process

Commercially available water glass solutions were diluted to 2.7 wt % with deionized water and stirred at 700 rpm (about 73 rad s⁻¹). Hydrochloric acid (8 M) was then added to adjust the pH to the desired value. The solution was stirred for about 10 min. Afterwards, the catalyst components (HY, kaolin) were added and stirred in the mixture for further 5 min. Spray drying of these slurries was carried out using a mini-spray dryer Büchi B-290 with a two-fluid nozzle. The samples were dried at an inlet temperature of 210°C, an outlet temperature between 80°C and 86°C, and a volumetric gas-to-liquid ratio of 427. Finally, the powder was calcined for 8 h at 650°C in dry air.

Binary and ternary mixtures were prepared in slurries for this work. The slurry formulation comprised a 1:1 (w/w) ratio for binary mixtures (HY-water glass and kaolin-water glass, cf. dry mass), and a 1:0.6 (w/w/w) ratio for the ternary mixture HY, water glass, and kaolin. The binary mixture comprised 39 wt % of HY or kaolin, 39 wt % of silicon dioxide, and 22 wt % of sodium chloride, and the ternary mixture comprised 31.7 wt % of HY, 18.9 wt % of kaolin, 31.7 wt % of silicon dioxide, and 17.7 wt % of sodium chloride from neutralization. The measurements for the samples prepared were repeated at least three times per sample.

2.3 Characterization of Slurry Properties

In the present work, two aspects of the slurries are characterized, namely, the rheological behavior and its relationship with the zeta potential. For the rheological study of the slurries, a Haake Viscotester iQ by Thermo Scientific was used with a double gap coaxial cylinder as measurement geometry (gap of 4.0 mm). The experiment comprised a stepwise strain rate increment from 50 to 1000 s⁻¹ in steps of 50 s⁻¹. The time required for this increase is 6 min, and each step takes 15 s. Afterwards, the system maintained a strain rate of 1000 s⁻¹ for 1 min to observe time-dependent shear flow properties (thixotropic behavior). The strain rate decreased stepwise back to 50 s⁻¹ (step size of 50 s⁻¹ and duration of 15 s).

Slurry apparent viscosity is determined from the collected shear stress data measured by the rheometer. Shear flow curves are fitted according to the Herschel-Bulkley equation [12]. The flow index obtained enables distinguishing Bingham (flow index of 1) and pseudoplastic behavior (flow index below 1). The analysis results can establish the minimum strain rate at which the viscosity is constant (10 % over the apparent viscosity at 1000 s⁻¹). All rheological measurements were performed at 25°C.

Furthermore, the zeta potential of slurries was determined with a Zeta-Sizer Nano by Malvern for each system and pH value. The sample was exposed to an electrical field and the ions’ movement was quantified under these conditions. Furthermore, a WTW LF96 electrode enabled comparing polarity changes of the particles (zeta potential) and the whole solution, including ions and solvent. The dispersant used in these measurements possesses a refraction index of 1.33.

2.4 Characterization of Solid-State Materials After Spray Drying

Particle size distribution of spray dryer products was determined by laser scattering with a Bettersizer S3plus from 3P Instruments. The equipment performed a wet dispersion procedure at 2000 rpm (209 rad s⁻¹) stirring and 200 W (26 kHz) ultrasonic for 120 s. Data acquisition and processing were carried out according to ISO 13320:2009. The dispersant used in these measurements possesses a refraction index of 1.33. The Mie theory was used for calculating particle size distribution from scattering data by [13].

In addition, scanning electron microscopy (SEM) was performed with an SU8020 SEM (Hitachi) equipped with a triple detector system for secondary and backscattered electrons (Uz = 2 kV). To acquire electron images, the samples were fixed directly on a double-sided adhesive carbon pad that was placed on an aluminum sample holder. To avoid any charge-up and chemical alteration during the measurements, the sample’s surface was coated with gold in an automatic rotary-pump coating system (Quorum Q150R ES).

3 Results and Discussion

Different slurries, i.e., water glass solutions and combinations with kaolin, zeolite HY, or both, are examined in this work. The rheological behavior of these systems is analyzed and compared considering the pH value of the slurry. The pH values chosen for this work were 10 (pH value of diluted water glass), 7 due to the observed gelation of the system, and 3 (to form a stable silica sol [14]). Furthermore, these results are related to the particle size distribution of the spray dryer samples. As a separate experiment, zeta potential and electrical conductivity of diluted slurries (1:100 vol.) with NaOH or HCl are quantified at different pH values to observe their peptizing behavior and peptization regimes.
3.1 Rheology and Peptization Behavior of Pure Water Glass

The slurry viscosity is presented in Fig. 1. The results clearly show that the apparent viscosity at pH 7 reaches values from 8 to 75 mPas. The precipitation and polymerization of silicic acid (gelation) are the reason for the viscosity increase. Additionally, pseudoplastic flow behavior is observed (index flow of 0.381). For this material, the gelation starts at pH 7–8 in accordance with Iler et al. [9]. Above 200 s⁻¹, the viscosity reduces to 20 mPas and the gel disappears. For quick acidification or higher strain rates during agitation, the pH value can be adjusted from 10 to 3 without gelation; both cases behave as a Bingham fluid. The slurry has a constant viscosity of 1 mPas at pH 3, which is lower than the value at pH 7.

![Figure 1. Viscosity curves of 10 wt% water glass in water at 25 °C and different pH values.](image)

Kind et al. [15] propose a surface charge modification of primary silica particles for a protonated (uncharged) surface at pH 2. With a further increase in proton concentration (pH < 2), the slurry tends to coagulate due to the reduction of repulsive forces at the particle surface. The redissolution rate of the gel at higher pH values (pH 3–10) increases with the [OH⁻] concentration in the slurry up to pH 10, where the silica solubility is high [15].

3.2 Properties of Binary Mixtures

The main component of an FCC catalyst formulation is the zeolite Y. For the mixture HY-water glass, the rheological behavior differs within the pH range observed. At pH 10, the zeolite does not interact with the water glass and the viscosity does not significantly alter. The zeolite particles do not coagulate, as observed with pure water glass. However, coagulation starts from pH lower than 8. The viscosity increment is slight (10% in comparison with pure water glass) after the addition of HY to the mixture at neutral pH due to poor zeolite particle distribution through the gelled silica. This effect is observed up to pH 5, where the gelation of the water glass is quick and the particles stick heterogeneously. Finally, the main difference in the system of pure water glass compared with the mixture of HY and water glass is the coagulation of the latter at pH 3 only after the addition of zeolite. This results from the destabilization of the slurry due to surface interaction of the aluminum of the zeolites with the silicates of the liquid phase [16].

The mixtures behave as a Bingham fluid in all pH ranges (flow index of 1), which means that the fluid shows a high flow resistance at low strain rates due to the agglomeration of the particles in the slurry. However, it reduces in a perfect reciprocal form during strain rate increment (Fig. 2a) due to the turbulent separation of the particles. The resulting effect of the viscosity change is the particle size increment after spray drying of the slurries, which reaches the spray dryer limit of 20 μm (Fig. 2c).

Polar groups in the zeolite surface promote the competitive adsorption of protons and silicate ions from the liquid phase. On the one hand, proton adsorption produces a buffering effect in the slurry, which was experimentally observed at lower pH values. On the other hand, silicate coverage produces a negative charge on the zeolite surface and repulsion among the particles, which is determined by the zeta potential (Fig. 2b). During acidification, the zeta potential is reduced below 10 mV (absolute value) at pH 8. Therefore, attractive forces of the system (van der Waals) overcome the electrostatic particle repulsion and the system coagulates. This effect becomes stronger at a lower pH value, which is coherent with literature reports about pH-dependent coagulation [17, 18].

A typical phenomenon of silicates is the electric conductivity reduction at pH 7 (Fig. 2b), which is caused by an ion strength reduction related to the neutralization process. In addition, gelation reduces the hydraulic conductivity, which is defined as the ease of a fluid to move through a system with solids [19]. This reduces the ion mobility in the slurry and thus its electrical conductivity.

Kaolin is another important component of the catalyst formulation. The addition of kaolin did not significantly alter the rheological behavior of the water glass slurries (Fig. 3). At pH 10, the interaction was low and the viscosity only slightly decreased (flow index of 1). In addition, kaolin has a weak influence on the slurry rheology at pH 7 (flow index increased to 0.73). The reason is similar to the mixture with HY zeolite, namely that the particles cannot disperse easily through the gel and the viscosity is not affected. Finally, there is no significant change at pH 3 and the viscosity slightly reduces. In comparison with HY, no pH buffering by kaolin was observed, which means that there is no significant interaction of the silicates with the kaolin particles at their surface. However, the slurry behaves nearly as a Bingham fluid (flow index of 1), as observed with pure water glass slurries. This can be attributed to the formation of independent micelles by water glass, which do not interact with the kaolin, as reported by Stempkowska et al. [20].

The agglomerates of kaolin-water glass reached 8 μm as the average size and the process is not as effective as with HY due to the low viscosity increment. This is attributed to a different surface polarity of kaolin compared with zeolite Y in its protonated form, observed through the increasing value of zeta potential (Fig. 3b). As a layered silicate, only a reduced number of polar silanol groups are present at the external surface to interact with silica from the water glass [21]. In addition, kaolin possesses...
more compact particles than zeolite Y, which reduce the contact of the surface with the silicates. Consequently, the silica binding to kaolin is less stable. There is also an ion strength reduction at pH 7 due to neutralization and precipitation of the silicates, as observed in the slurries with HY (Fig. 3b).

Figure 2. Influence of pH for slurries with HY-water glass by means of (a) viscosity, (b) zeta potential, (c) particle size distribution of the final product after spray drying.

Figure 3. (a) Viscosity, (b) zeta potential, (c) particle size comparison for the kaolin-water glass system at different pH values.

### 3.3 Properties of Ternary Mixtures

For the mixture of all three components of interest, there was a slurry viscosity increment, which looks like a superposition of the binary effects. At pH 10, there was no viscosity increase due
to a high solubility of silica and no coagulation of water glass (Fig. 4a). Moreover, viscosity only increases due to the addition of HY at pH 3. Nevertheless, there is a more effective coagulation at pH 7, which seems to be a combination of the aggregation of HY, the gelation of water glass, and the chemical binding of HY with silica. In all cases, the fluid showed a Bingham behavior and no pH changes were observed during agitation. Ion strength is also reduced at pH 7 by neutralization and silicate precipitation (Fig. 4b).

The results in comparison with binary mixtures demonstrate that effective binding only occurs in the presence of zeolite Y. Kaolin cannot coagulate the mixture (see Fig. 3a) due to its low phase contact with the binder and limited adsorption of silicates at their surface. However, zeolite Y has a larger contact area with the binder and adsorbs more binder particles at its surface. This modifies the colloidal behavior of the zeolite particles, which change their surface charge with increasing adsorption of negative binder particles. As a result, the slurry destabilizes at neutral to acid pH values. Therefore, the rheological behavior of the ternary mixture is dominated by the interaction of the HY with the binder.

3.4 Influence of Mixing Speed on the Peptization of Slurries with Water Glass and HY

On the one hand, the surface charge of the particles determines the magnitude of the electrostatic repulsion force. On the other hand, the particles are attracted by van der Waals forces, which depend on the particle size and material [22]. If attractive forces surpass the electrostatic repulsion, the slurry coagulates. For particle separation, external mechanical forces are introduced. From rheology, it is well known that agitation efficiency is coupled with the fluid shear stress. The mechanical stress applied to the liquid is a variable that depends on the velocity gradient of the fluid.

Considering the geometry of a stirred tank, Sánchez et al. [23] proposed the following model for determining the shear rate in turbulent flow:

\[
\dot{\gamma} = \left( \frac{4N_P d_i^2}{\pi^3 K} \right) \frac{1}{N_i^{1/3}} = mN_i^{1/3}
\]  

(1)

and

\[
N_P = \frac{2\pi M}{\rho N_i^2 d_i^3}
\]

(2)

The last equations use parameters of the Herschel-Bulkley model, which is described in Eq. (3) for the fluid shear stress \( \tau \):

\[
\tau = K\dot{\gamma}^\mu + c
\]

(3)

The equilibrium of forces as a new model, i.e., electrostatic repulsion, van der Waals attraction, and mechanical separation, defines the critical gel formation in the system with a linear, but slow coagulation rate. Consequently, the electrostatic repulsion absence may be compensated by mechanical shear to sta-
bilize a coagulating slurry and to control the coagulation period [24].

\[
\frac{F}{R_{\text{eff}}} = \frac{A}{60} + \frac{4\pi \varepsilon_0 \varepsilon_{\text{r}} K^2 e^{-\frac{e^2}{kT}}}{\text{Mechanics}} + \frac{c_0}{\text{Van der Waals}} \implies 0 \tag{4}
\]

Shear stress is stirring rate-dependent. During agitation of the slurry, a vortex pattern was observed. This forms turbulent regions with high strain rate and stagnant regions with reduced shear [25]. For a pseudoplastic fluid, e.g., the slurries of the present work, this means a heterogeneous viscosity distribution in the stirring recipient, where the viscosity is higher in the stagnant region. A similar phenomenon has been studied for stirred tanks [26], where stagnant regions produce local viscosity changes. These local viscosity gradients generate slugs in the liquid feed for the following spray dryer step. Considering that viscosity determines the droplet size and intermittency of spraying [27], slugs produce a heterogeneous particle agglomeration during drying. This phenomenon is reduced at higher stirring rates due to the increase of the strain rate in the mixing recipient (Fig. 5).

The minimum speed rate for homogeneous size distribution after spray drying is 900 rpm (94 rad s\(^{-1}\)) at pH 3 from experiments. At higher rates, the apparent viscosity only slightly decreases. Below this critical mixing speed, the viscosity of the slurry sharply increases, which causes uncontrolled agglomeration.

By modeling the system force balance, the minimum agitation speed for an increment of max. 10% of the minimum viscosity was 1020 rpm (107 rad s\(^{-1}\)), which fits well with the experimental value. The slight deviation can be attributed to the calculated strain rate as an average value and the choice of the stability criterion for the viscosity.

The calculations originate from the Derjaguin-Landau-Verwey-Overbeek (DLVO) theory [24] and include the following side conditions:

- Interacting silica particles from water glass show a diameter of approximately 1 nm (silicate).
- The viscosity of gelled slurries is focused due to the drastic viscosity increment.
- To establish a minimum strain rate for the stability of the slurry a 110% value of the minimum viscosity was used. The critical strain rate value defines the force equilibrium.

### 3.5 Determination of Peptization Regimes for Control of the Spray Drying Process

As a major assumption from Sects. 3.2 and 3.3, the coagulation behavior of the binary mixtures should superimpose in ternary or multicomponent mixtures as the basic attractions. Moreover, the critical absolute value of zeta potential for stable slurries is 20 mV from theory, which confirms observations for slurries without additional agitation (see Sects. 3.2 and 3.3). If mechanical force is added, the distance among particles can be established. For this calculation, an imbalance between the forces due to peptizing is equilibrated by the mechanical input of the mixer at the critical agitation speed. The calculated distances of particles during agitation at 900 rpm are given in Tab. 1. Further information about zeta potential-depending repulsion and particle distance relation can be found in the Supporting Information (Fig. S1).

#### Table 1. Calculated equilibrium distances during agitation at 900 rpm (94 rad s\(^{-1}\)) and pH 3.

| Bond          | Equilibrium distance [nm] |
|---------------|---------------------------|
| Silica-silica | 8                         |
| HY-HY         | 360                       |
| Kaolin-kaolin | 590                       |
| HY-kaolin     | 480                       |

**Figure 5.** (a) Variation of viscosity and shear mechanical forces of the system HY-water glass with agitation in dependence of the rotation speed at pH 3. (b) Particle size distribution dependence of agitation speed for the system HY-water glass.
The results of the particle distance calculation show a higher bond distance for kaolin-kaolin in comparison with HY-HY, caused by lower attractive forces among the kaolin particles. At a zeta potential below the critical value, the particles in the slurry should form a gel. However, kaolin surprisingly does not coagulate in acidic media. An explanation is given by its morphology (Fig. 6), as kaolin particles show high density and packing behavior, and therefore they need more energy to form stable bonds. Furthermore, the surface species of HY are mostly populated by silanol groups, which may cause chemical bonding and gelation by condensation after physical attraction. In addition, kaolin mostly has non-condensable silicon dioxide structures that may not allow adsorption. Therefore, the kaolin gelation is less probable.

From theory and experiments, pH value, zeta potential, and agitation can control particle shaping, and basic slurries (pH > 9) are not suitable due to the high solubility of silica. A regime of neutral media (pH 4–8) leads to overabundant and uncontrolled coagulation of silica and HY. Only acid slurries (pH 2–3) are suitable to control the coagulation of water glass, and the particle size distribution of catalyst prototypes after spray drying. At pH 3, a model-supported reference value is given for the devices and slurries used. It can be easily adjusted to other setups by force balance and a few agitation experiments.

Therefore, the particle size controlling method for spray drying products comprises the following steps. Mixing of catalyst components (zeolite Y, kaolin, water glass) results in a pH of 10. Afterwards, quick acidification to pH 3 is combined with fast agitation to maintain the slurry stability. Finally, the slurry has to be stirred vigorously during the whole process to produce a narrow particle size distribution of products.

Given that this method establishes a controlled particle size production, it can be applied for the optimization in the production of other catalysts that use silica species as a binder or support material. A good example is the acryl nitrile production, whose catalysts are supported in silicates [28, 29]. In addition, this control method is optimal for use in the formulation of FCC catalysts from recycled materials. This application has stronger relevance in the adaptation of FCC to the new market requirements. Finally, another important application of this method is adsorbents based in silica. Indeed, silica gel is widely used as an adsorbent and its performance depends on the particle morphology [30, 31].

4 Conclusion

With a combined approach of fundamental forces and experimental observations, it is possible to understand and control the basic surface chemistry of catalyst particle binding by silica coagulation by consideration of rheological phenomena with zeolite Y. Therefore, the main results are summarized as follows:

- The surface (electro-)chemistry and stability during silica agglomeration (from water glass), kaolin and zeolite Y is explained by a critical zeta potential of 20 mV (absolute value) from theory and the experiment. Below the necessary surface polarity, an uncontrolled physical aggregation is observed. For high-concentrated surface hydroxyls (HY but not kaolin), an uncontrolled condensation leads to an uncontrolled coagulation.
- Van der Waals attraction and electrostatic repulsion are sufficient to quantitatively describe and estimate the peptization behavior of silica-based slurries.
- The resulting model describes the peptization behavior in the presence of mechanical forces from agitation. It establishes a critical mixing speed of 1020 rpm, which is in close proximity to 900 rpm at pH 3 from the experiment. Finally, particles up to 20 μm (spray dryer limitation) are produced. Therefore, a particle size controlling method comprises vigorous stirring and quick acidification of water glass slurries to avoid undesired gelation in the process.

The novelty of the investigations compared with the silica peptization chemistry by Iler et al. [9] and the mechanical behavior by agitation by Kind et al. [15] lies in the combined and theory-supported chemical gelation control of silica-based slurries including solid particles (HY, kaolin). The reported results have a fundamental theoretical basis and should therefore be scalable for equipment with higher capacity.
Supporting Information

Supporting Information for this article can be found under DOI: https://doi.org/10.1002/ceat.202000480.

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Symbols used

\[ A \] [J] Hamaker constant
\[ c \] [Pa] ordinate value in the Herschel Bulkley model
\[ d \] [m] tank diameter
\[ d_i \] [m] impeller diameter (stirrer)
\[ F \] [N] force between particles
\[ h \] [m] distance among particles
\[ K \] [-] viscosity coefficient in the Herschel Bulkley model
\[ m \] [-] relation coefficient for turbulent flow in stirred tanks
\[ M \] [Nm] minimal stirrer torque
\[ N \] [rad s\(^{-1}\)] stirrer rotation speed
\[ N_p \] [-] stirrer power number
\[ R_{eff} \] [μm] particle effective radius

Greek letters

\[ \alpha \] [kg m s\(^{-1}\)] proportionality constant between force and strain rate
\[ \gamma \] [s\(^{-1}\)] strain rate
\[ \varepsilon \] [F m\(^{-1}\)] vacuum permittivity
\[ \varepsilon_0 \] [-] dielectric constant
\[ \zeta \] [V] zeta potential
\[ \eta \] [Pa s] apparent dynamic viscosity
\[ \kappa \] [m\(^{-1}\)] inverse Debye length
\[ \rho \] [kg m\(^{-3}\)] fluid density
\[ \tau \] [Pa] fluid shear stress

Abbreviations

FCC fluid catalytic cracking
SEM scanning electron microscopy

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