

Dispersion Properties of Colloidal Graphite

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Colloidal properties of dispersed systems consisting graphite particles and kind of additives were investigated. Experiments have been conducted using carboxymethyl cellulose (CMC) and polyvinyl alcohol (PVA) as additives and water, alcohol and acetone as dispersions media. The effect of additives, binder concentrations, dispersions media and graphite contents on colloidal properties of graphite suspensions were mainly discussed by means of sedimentation behavior, rheology and turbidimetry measurements. Results showed that when graphite have been dispersed in water consisting 10 % mass alcohol and 0,1 mass % carboxymethyl cellulose or 0,15 mass % polyvinyl alcohol, the best dispersion has been obtained.

1 Introduction

Colloidal suspensions of carbon have many industrial applications. They are now being studied for use in conducting composite materials, liquid electro photo graphic toners, pigments in inks and paints, rubber reinforcements and fillers, electrodes and conducting films [1]. The surface properties of dispersed particles in suspensions are important for their dispersion properties. Many studies on the properties of colloid dispersed systems have been reported [2–5]. The

structure of the dispersion is highly dependent on the process of formation of the flocs, size of the dispersed particles, wettability to dispersion medium, and strength and nature of attractive forces [3–5]. Colloidal graphite is a fine graphite powder which suspended in a liquid carrier (such as water and/or alcohol), together with a small amount of a binder [6]. Graphite based suspensions can be successfully prepared in an aqueous system. This was accomplished by preparing a stable slurry using organic additives, namely, carboxymethyl cellulose (CMC) and polyvinyl alcohol (PVA). The highly conductive graphite particles are very hydrophobic in natural state and can only be suspended with vigorous agitation [7]. To overcome this, a polymeric anionic binding should be used to coat graphite particles as to cause the particles to repel each other rather than link together via the attractive Van der Waals forces. Once the graphite is coated with the binder, the colloid can now be easily dispersed in water and is sterically stable [7]. In the present study the effect of binder concentrations, dispersions media and solid volume fractions on dispersion proper-

Table 1 Specifications of graphite powder as starting material

Density	2,21 g/cm ³
Water content	0,33 %
Ash content	5,36 %
Particle size (d ₅₀)	8,28 μm
Specific surface area	1,56 m ² /g

ties of graphite in colloidal aqueous suspension, determined by turbidimetry curves and rheological behavior of suspensions. Additionally, sedimentation behavior after 3 months incubation time at room temperature was investigated.

2 Experimental Procedures

The scale-shaped graphite powder with an average particle diameter of < 10 μm (MEGA plant, Germany, carbon content 96,5 %) was used as starting material. The rheology measurements of graphite suspensions were made using a cone and plate viscometer

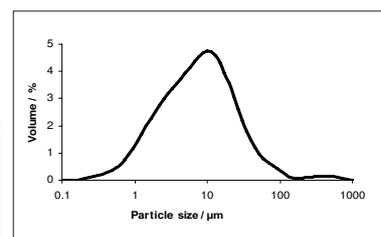


Fig. 1 Particle size distribution of graphite powder as starting material

of Physica MRC300 (Germany) for shear rates from 0 to 2600 1/s. Particle size distribution of graphite was studied by laser diffraction microanalyser Master size 2000 (Malvern Co.). Table 1 presents the specifications of graphite powder used in this research. Fig. 1 shows particle size distribution of graphite. The value of specific surface area and density were determined from Quantasorb

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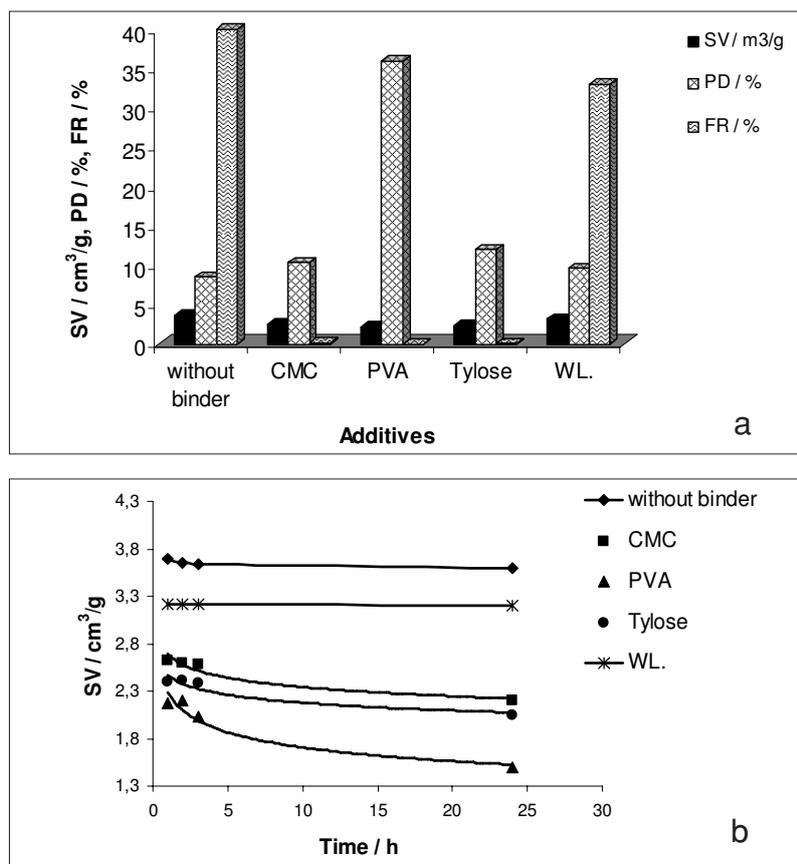


Fig. 2 a) SV, PD and FR of graphite suspensions with different kinds of binders (0,1 mass %), b) SV of graphite suspensions after 24 h setting aside

BET and Helium pycnometer (*Micromeritics Accupay 1330*). Water and ash contents were determined by using ASTM C562-91 and C561-91 respectively. Turbidity was determined by using the 2100N (Hach Co.). Colloidal

graphite suspensions were prepared by mixing graphite powder (20 wt%) dispersed in a solution of binder (normally 0,1 mass %). Carboxymethyl cellulose (CMC), poly- vinyl alcohol (PVA), Tylose (hydroxy ethyl cellulose) and dishwash-

ing liquids (Pakshoo company, Iran) (WL) were used as additives. All suspensions were prepared by deionized water, prepared by mechanical mixer (IKA RW.20n, IKA) during 15 min with 1100 rpm. After mixing, the samples were placed into cells for sedimentation volume and rheology parameters measurements. After agitation of graphite and binder solution for 15 min, the suspensions were set aside for 10 min. The graphite sedimentation layer height and the amount of floating graphite were measured through gathering them by a spoon and drying at 100 °C, and sedimentations volume SV (cm³/g), packing densities of sediment layer (PD) (%) and floating ratio FR (%) calculated as follows:

SV = volume of sedimentation layer/ weight of sedimentation layer; PD = 100/(SV × graphite true density); FR = weight of floating graphite/ total weight of graphite [8-11]. Sedimentations volume, turbidimetry and rheology parameters of graphite powder in aqueous suspensions were measured at fixed temperature T = 298 K. To obtain reproducible results, all measurements (3 samples for each test) were carried out by first suspension preshearing to break up any large aggregates (1-upward shear rate ramp, in t = 100s: $\dot{\gamma} = 0$ to 2600 1/s, 2-Rest phase, for t = 100 s: $\dot{\gamma} = 0$). Every test was followed by 50 points measurements.

3 Results and Discussions

The aqueous wettability of no binder suspensions and binder suspensions of graphite were compared. SV and FR should ideally be low and PD high for good wettability. Fig. 2 shows the SV, PD and FR of graphite suspensions with different kind of binders. It can be seen that suspensions were prepared with PVA, Tylose and CMC are the best dispersed suspensions. Increasing binders in graphite suspensions caused the SV to decrease to 1,8 cm³/g and the FR to zero, indicating that the wettability of the graphite was improved. The effect of PVA binder on wettability and dispersion behavior was reported better than

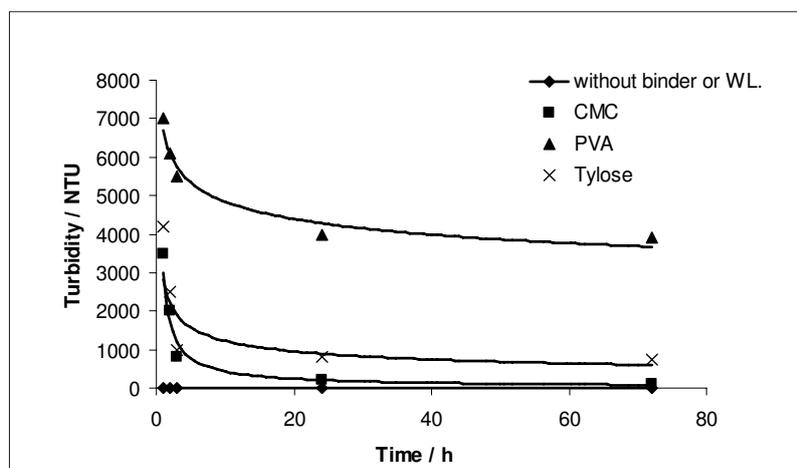


Fig. 3 Turbidimetry of various suspensions as a function of time with different kinds of additives (0,1 mass %)

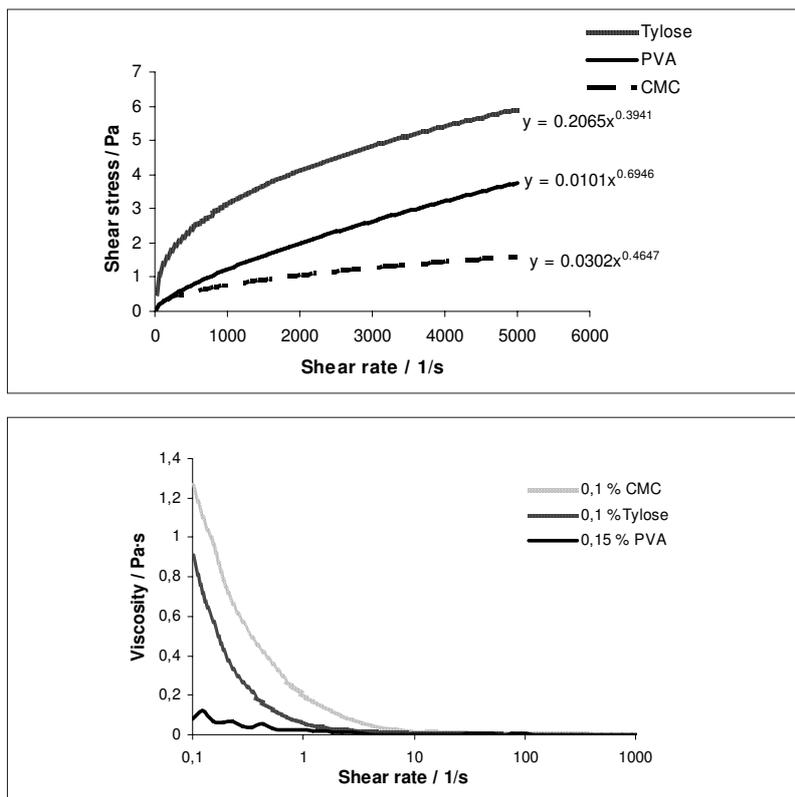


Fig. 4 a) Flow curves of graphite powder in water with different amounts of binders (0,1 mass %), b) Viscosity versus shear rate of graphite powder in water with different amounts of binders

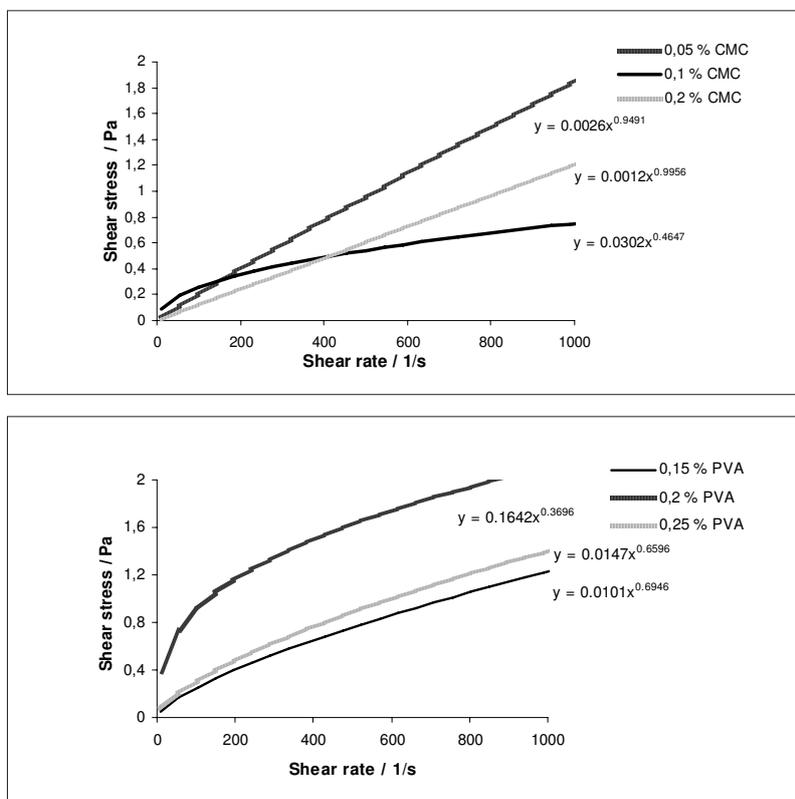


Fig. 5 Flow curves of aqueous graphite powder in water at different concentrations of a) CMC and b) PVA

CMC [12, 13].

However, from the SV measurements can be seen that washing liquids (WL) only decreased the SV volume of graphite slightly; even though the WL content in suspension is high. This result maybe attributable to poor quality of WL additives in dispersing graphite. As is clear from these graphs, PVA, Tylose and CMC improve the water wettability of graphite, although PVA binder appears to be more effective. Fig. 3 shows the turbidimetry of suspensions during 72 h. In turbidimetry the amount of light passing through a solution is measured. The higher the turbidity, the smaller the quantity of light transmitted. Turbidity is reduction in transparency of a continuous phase due to light scattering and absorption caused by a dispersed phase. Thus it is a function of size and dispersed species concentration [14, 15]. Fig. 3 also improves earlier results. All binders improved graphite dispersion, although the graphite suspension with PVA showed the best dispersion, a result which may be attributable to its high -OH functional groups.

The poor aqueous wettability and dispersion behavior of graphite is associated with presence of few hydrophilic functional groups, such as -OH or -COOH, on its surface [9]. To improve wettability and graphite dispersion in water, various methods have been investigated, including using surfactants, coating graphite surface with other materials, and making graphite micro pellets or briquettes. Surfactant additions have been used to improve the aqueous wettability and dispersion of graphite since they can increase the water affinity. This approach is easy to use at relatively low cost, but its drawback is that the improvement in wettability and dispersion behavior is limited, so it is still difficult to incorporate a large amount of graphite. Suspensions were prepared with PVA, Tylose and CMC as binders, have been chosen for rheological measurements. Rheological properties of suspensions are very sensitive to their structural organization, which is controlled by the amount of binders. Fig. 4 shows viscosity and shear stress / shear rates of graphite suspensions contain-

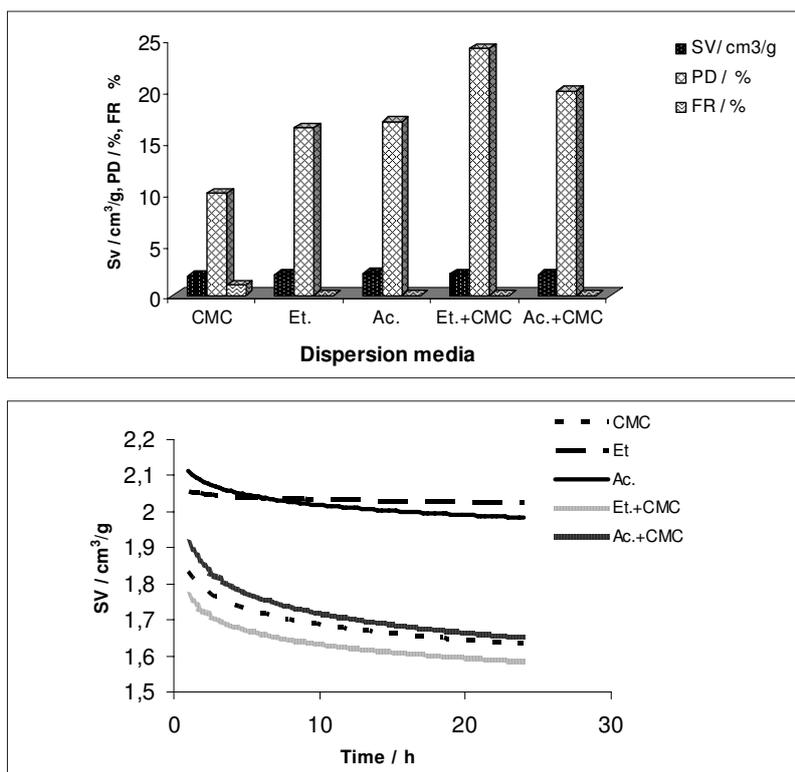


Fig. 6 a) SV, PD and FR and b) SV after 24 h setting aside of graphite suspensions prepared with alcohol or acetone as dispersion media

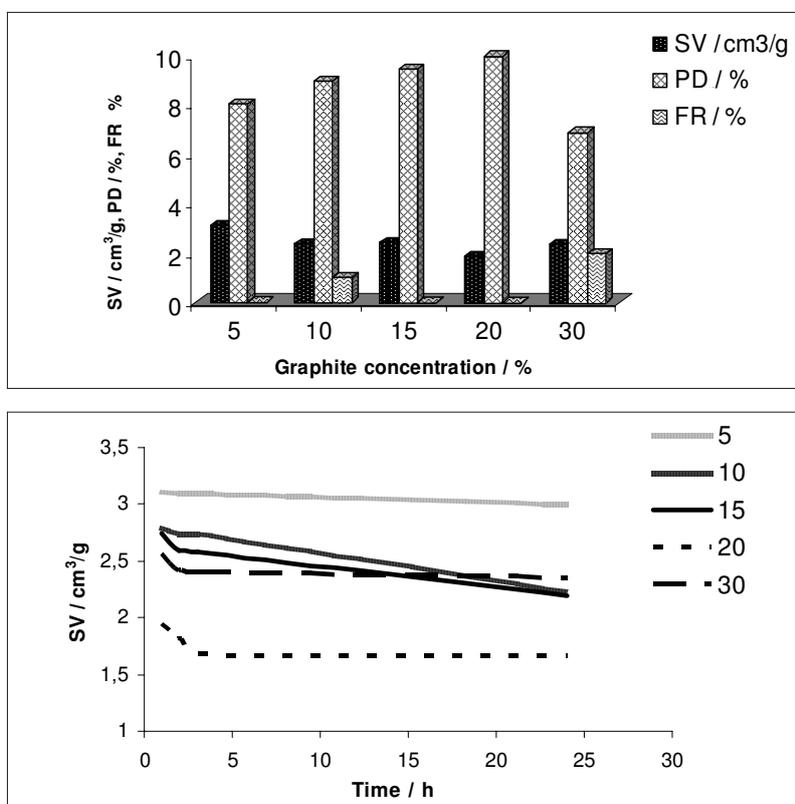


Fig. 7 Sedimentation behavior of aqueous suspensions at different concentrations of graphite powder a) SV, PD and FR and b) SV of graphite suspensions after 24 h setting aside

ing mass fraction of 20 mass % solid with different binders (0,1 mass-%) and Fig. 5 shows rheological curve of graphite in water at different CMC and PVA concentrations. In the absence of any surfactant, graphite particles sedimented even at very small volume fractions. It reflects existence of a strong aggregation between graphite particles. In contrast, in the presence of a surfactant, a well-dispersed graphite suspension is observed; due to decrease of particles aggregation. Increasing the dispersant concentration in solutions leads to an increased density of the surface-adsorbed layer, thereby reducing the attractive interactions between particles (lower shear stress). The binder molecules form the stabilizing layers on the surface of the graphite. All graphite suspensions have shear-thinning behavior. In general, the dispersion state increased with increasing CMC and PVA content up to 0,1 mass % and 0,15 mass % respectively since the surface active groups on graphite surface also increased.

Fig. 6 shows the sedimentation behavior of graphite suspensions prepared with 10 mass % alcohol (Et.) or acetone (Ac.). It can be seen that alcohol or acetone improves the dispersion of graphite suspensions (lower SV and high PD), although the alcohol and CMC appears to be more effective. Different amounts of dispersions media were investigated; however the best results are reported in this work. The SV of suspensions are continuously decreased and the PD is increased up to 20 mass % graphite with graphite concentration increasing (Fig. 7). Fig 8 shows the turbidimetry of graphite suspensions. The best curves are 20 mass % and 30 mass % suspensions. However the turbidity of other suspensions were very negligible. The shear stress begins to show shear-thinning only at graphite concentrations > 20 mass % (Fig. 9). As graphite concentration increased, SV values of suspensions decreased. The observed behavior for SV and shear stress evidences existence of house card structures in suspensions which reflects the rheological properties of suspensions. House of card structure of dis-

persed systems may display different rheological properties, depending on volume fraction of solid phase, geometry of particles and presence chemical additives. In concentrated suspensions, house card structure may arise more stability of suspensions.

4 Conclusion

Experimental data on colloidal properties of aqueous graphite suspensions as function of solid volume fraction, liquid phase and binder concentrations were studied. Because of high hydrophobicity of graphite, particles tended to aggregate in water suspensions and sediment, but addition of a surfactant allows to achieve fine adjustment of aggregation processes through changes in surfactant concentrations. To improve graphite suspensions stability, it is necessary to high agitation and use CMC and PVA as binders. Organic binders played a key role as a thickening agent to prevent graphite particles from settling out during processing. It was found that organic binders also contributed to the development of surface electric charge on graphite particles. Furthermore the graphite particle size must be at most 10 μm . The surfactant molecules may form coating around particles of graphite, and it results in decrease of shear stress with increase of shear rate or solid concentration. The best dispersion can be observed in 0,1 mass % CMC or 0,15 mass-% PVA with 10 mass % alcohol.

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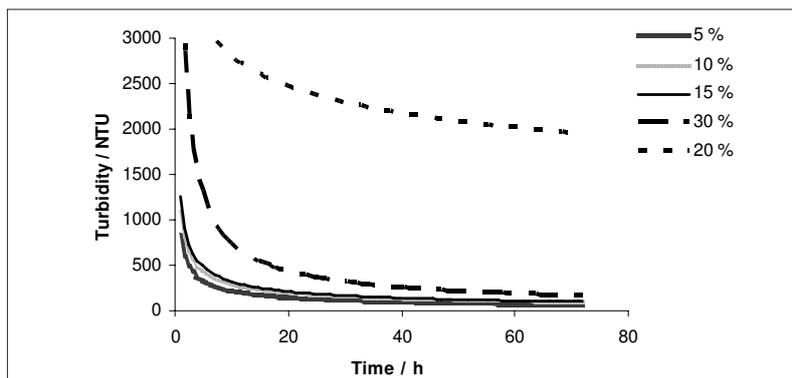


Fig. 8 Turbidimetry of suspensions at different concentrations of graphite powders

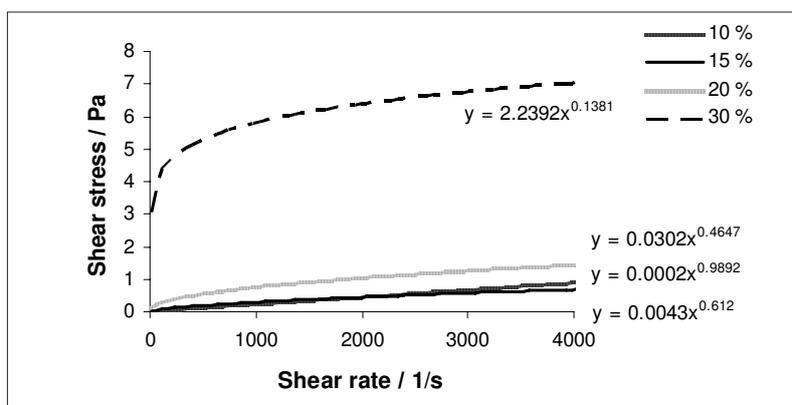


Fig. 9 Flow curves of graphite powder in water at different concentrations of graphite

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