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Influence of capsule dosator wall texture and powder properties on the angle of wall friction and powder-wall adhesion

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Summary

The influence of capsule dosator wall texture and powder properties on the angle of wall friction and powder-wall adhesion was investigated using a modified annular shear cell. Powder-wall friction studies were conducted for different size fractions of five pharmaceutical excipients and two dosator wall materials of defined textures. Results indicate that the angle of wall friction (Φ) and powder-wall adhesion are functions of the powder material, its particle size and the dosator wall roughness. For a particular excipient, smaller values of Φ are generally observed for coarser size fractions and a smoother wall surface.

Introduction

During the filling of powders using automatic capsule machines operating on the dosator nozzle system, stresses are generated at the contacts between the nozzle wall and powder mass within the dosator. Powder retention ability and the ease of its subsequent ejection into an awaiting capsule body may be greatly influenced by powder-wall friction (Jolliffe and Newton, 1983).

Various methods have been used in the measurement of powder-wall friction. Jenike (1961) first used a shear box tester to measure the kinematic angle of friction between a solid and a wall material. Jolliffe and Newton (1983) used the Jenike technique to measure the angle of wall friction between a powder (lactose) and the dosator nozzle wall material. Their work pointed to the importance of the angle of wall friction and the interaction between the powder and the nozzle wall surface in influencing the retention of the powder within the nozzle.

The angle of wall friction for powders is determined as the angle between the wall yield locus and the abscissa. The wall yield locus is evaluated from the relationship between the shear stress required to cause sliding of a layer of powder in contact with a wall or substrate under a compression stress normal to the plane of the substrate and the compression stress (Treasure, 1966). Similar equipment had been used by Miller and York

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(1985) in the assessment of the friction between a lubricant powder and a flat surface. A modified annular shear cell similar in design to that of Carr and Walker (1967) was used to study the frictional properties of lubricants by Baichwal and Augsburger (1985).

Wall-friction studies have also been undertaken by pulling a plate of wall material across a powder compact under pressure (Strijbos, 1976, 1977; Hirai and Okada, 1982; James and Newton, 1983; Wagner, 1983). The use of instrumented punch and die assemblies to measure friction coefficients during powder compression and ejection has been reported by Hölzer and Sjögren (1981a,b) and Kikuta and Kitamori (1983).

This paper describes the use of a modified annular shear cell to study the powder wall interaction between different size fractions of five pharmaceutical excipients and two capsule dosator nozzle wall materials of defined textures. The influence of wall texture and powder properties on the angle of wall friction and powder-wall adhesion was investigated.

Materials and Methods

Materials

Different size fractions of microcrystalline cellulose, Avicel PH101, pregelatinised starch (Starch 1500), calcium carbonate, maize starch and lactose monohydrate, fractionated and characterised as described previously (Tan and Newton, 1990), were used in the present work.

Methods

Surface texture measurement

The most commonly used parameter to describe surface texture is the average roughness, R_a (or the centre line average roughness, CLA). This is defined as the arithmetical average value of the departure of the profile above and below the reference line (centre or electrical mean line) throughout the prescribed sampling length (BS 1134, 1972).

R_a values are commonly measured by profilimetry. This is by means of an electrical integrating stylus instrument with a diamond stylus mounted on a pick-up arm which is drawn across the specimen surface at a steady rate. The vertical movement of the stylus as it follows the surface irregularities produces an electrical signal proportional to the local height of the surface. These electrical signals can be amplified to various ranges depending on the magnification required. In addition to producing a magnified trace of the surface profile, the instrument is usually capable of calculating and producing a printout of the R_a values and associated surface roughness parameters.

In the present work, a Surfcom 550A surface finish measuring instrument (Advanced Metrology Systems Ltd, Leicester, U.K.) was used to assess the dosator nozzle wall texture of two different stainless-steel surfaces (St and Mt). Surface roughness measurements were made both along the radius and perpendicular to the radius of the plates. The determinations were undertaken by Advanced Metrology Systems Ltd. Scanning electron micrographs of the surfaces of plates were also taken.

Powder-wall friction

The apparatus used to study powder-wall friction was essentially an annular shear cell (Technigraphic Bristol Ltd, U.K.) similar in design to that of Carr and Walker (1967). Details of the cell and its use for evaluating powder failure properties are described elsewhere (Tan, 1987). For the present work, the shear cell lid was replaced with an annular stainless-steel plate of outer diameter 12.64 cm and inner diameter 7.68 cm. Two specially prepared, annular plates (St, turned and polished; Mt, turned) of defined surface roughness (see Surface texture measurement) were used. The surface textures were similar to those found in dosator nozzles used in capsule machines.

The powder sample for testing was prepared in the same way as for powder failure studies (Tan, 1987). The lid with the annular plate was carefully lowered onto the powder bed and then sheared to failure against the wall surface. Subsequent points on the wall yield locus were obtained by shearing the powder sample to failure at reduced normal loads without prior reconsolidation of the powder between measurements. The weight of the lid plus the annular plate constituted the maximum consolidating load imposed upon the sample.

The experiment was carried out on all the powder systems using the two different annular plates. Determination of each point on the wall yield locus was repeated at least twice and the plate was carefully cleaned and dried before testing a new sample.

Angle of internal friction (δ)

The results reported are taken from Tan and Newton (1990).

Results and Discussion

Surface texture measurement

The values of R_a for plate Mt (Table 1) are consistently higher than those of plate St, both radially and circumferentially along the plates. Whilst the R_a values of plate St are quite similar (approx. 0.1 μm) along both axes, those of plate Mt are markedly different. The value of R_a radially (1.18 μm) is approx. 4-times that determined circumferentially (0.42 μm). Examination of the surface profile traces obtained (Fig. 1a–d) confirms the marked differences between the two plates. The peak heights and troughs for plate Mt are much more pronounced and are also more variable than those of plate St. Fig. 1c and d also shows the marked difference in the surface profiles of plate Mt when measurements are made on different axes along the plate.

TABLE 1

Arithmetical mean deviation (R_a) values of St and Mt plates

Axis of measurement	R_a (μm), x (±S.D.)	
	Plate St	Plate Mt
Radially	0.13 (±0.1)	1.18 (±0.06)
Circumferentially	0.10 (±0.01)	0.42 (±0.18)

St, 'smooth' textured plate; Mt, 'medium' textured plate; cut off, 0.80 mm; traversing length, 8.00 mm; magnification: vertical trace, ×5000; horizontal trace, ×20; x, mean value (of three measurements); S.D., standard deviation.

Scanning electron photomicrographs taken of the two plates' surfaces (Fig. 2) further confirm the rougher surface profile of plate Mt compared to plate St.

Powder-wall friction

Typical graphs of shear stress plotted against normal stress on the two different textured annular plates (St and Mt) for two of the powder systems (calcium carbonate, C and maize starch, M) are shown in Fig. 3. In all cases, linear graphs having a correlation coefficient, $r > 0.99$, are obtained. These straight lines often pass through the origin, but in certain cases, intercepts at low values on the shear axis are observed. The angle of wall friction Φ is determined from the tangent of

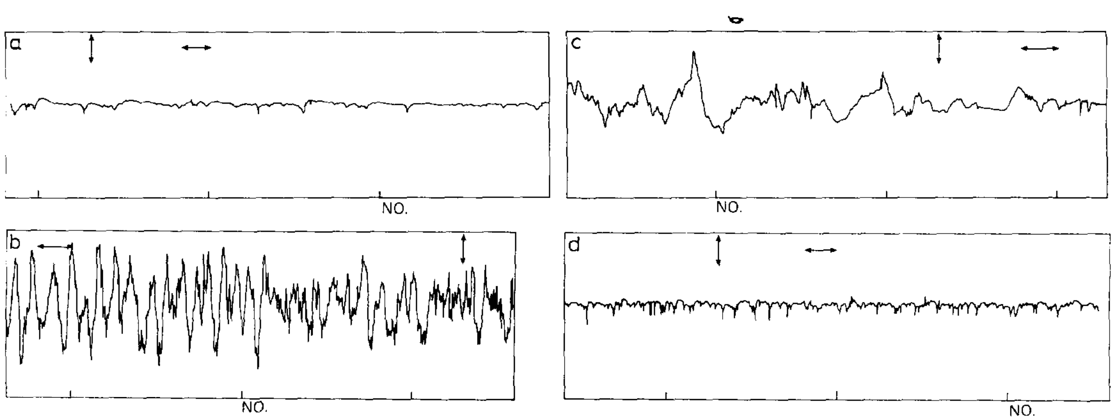

Fig. 1. Surface profile traces of annular plate. (a) St, circumferentially along the plate. (b) St, radially along the plate. (c) Mt, circumferentially along the plate. (d) Mt, radially along the plate. Vertical scale, ×5000; horizontal scale, ×20.

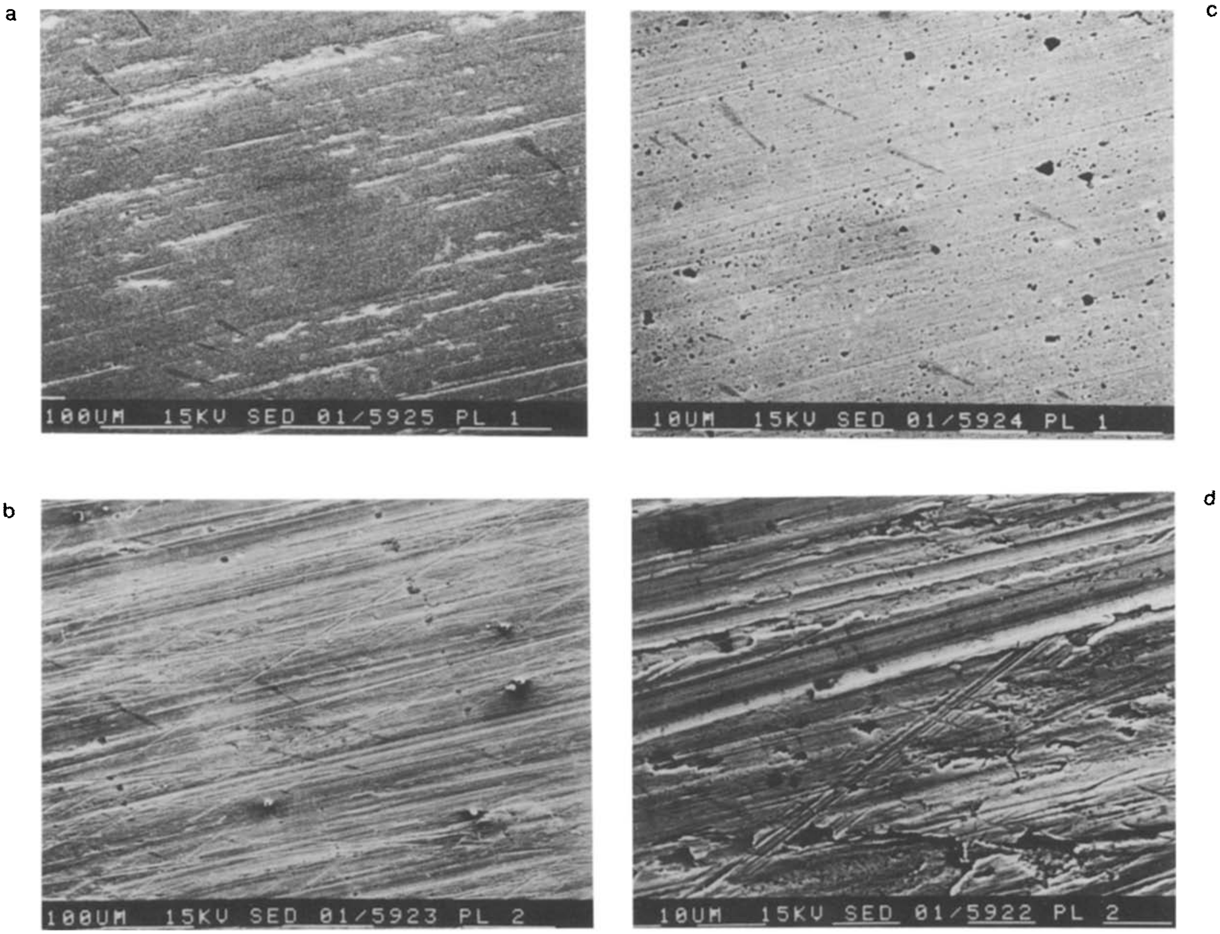

Fig. 2. SEMs of annular plate surfaces St (a,c) and Mt (b,d).

the slope of the wall yield locus. Values of Φ for the different powders are listed in Table 2. Plots of tan $\Phi(=\mu_d)$ as a function of log of the ratio of the particle size to wall roughness are shown in Fig. 4a and b.

It is observed (Table 2) that for both plate surfaces, Φ generally decreases as the particle size of the material increases. Values of Φ for the smoother surface (plate St) are consistently smaller than the rougher surface (plate Mt). Also, powder-wall adhesion (seen as a positive intercept of the wall yield locus on the shear axis) appears to be a function of the powder material, its particle size and the wall texture (e.g. Fig. 3a–d). For both plate surfaces, low values of Φ are shown by powders A2, A3 and M2, while higher Φ values are observed for L1, L2 and L3 (see Table 2).

The difference in the values of Φ shown by the present powders may be satisfactorily explained by the postulate of Strijbos (1976, 1977), who studied the powder-wall friction of ferric oxide powders on cemented tungsten carbide wall. He suggested that the coefficient of dynamic power-wall friction, μ_d $(=\tan\Phi;\ \Phi$ in $^\circ)$ could be related to two dimensionless parameters, viz. the ratio of mean powder particle size to wall roughness (d_p/R_w) and the ratio of powder particle hardness to wall hardness (H_p/H_w). The influence of the orientation of wall grooves also needs consideration in the measurement of μ_d.

In cases where $d_p/R_w < 1$, fine particles tend to be trapped in the surface irregularities. Thus,

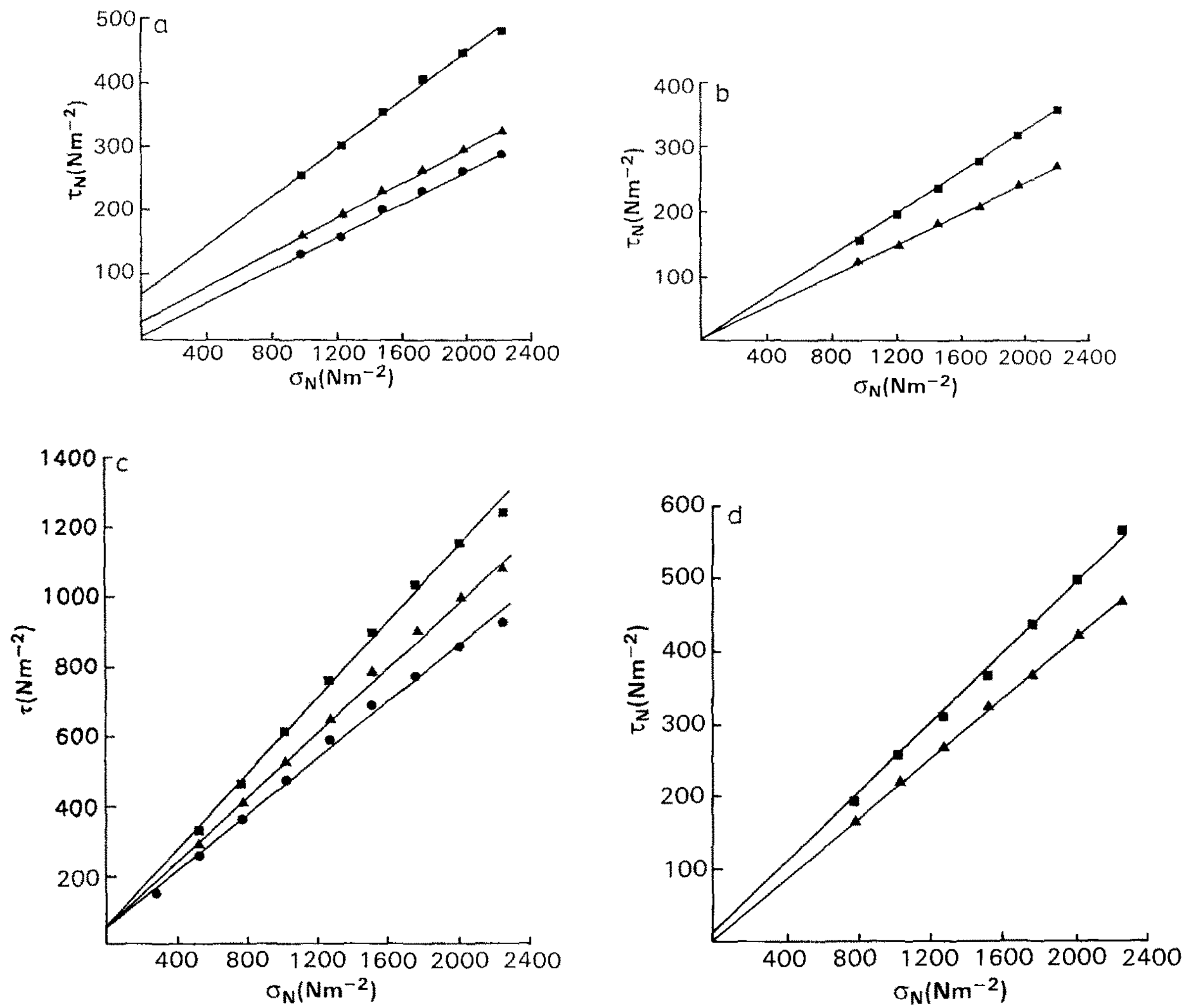

Fig. 3. Shear stress (τ) as a function of normal stress (σ_N) for calcium carbonate (a, c) and maize starch (b, d) on the annular plate St (a + b) and Mt (c + a). (a, c) ■, C1; ▲, C2; ●, C3. (b, d) ■, M1; ▲, M2.

during experimentation, the friction measurements obtained represent both powder-wall friction and powder-powder friction due to the presence of this 'sticking' layer of powder at the wall. With increasing powder build-up during shearing, the angle of wall friction may in fact approach the angle of effective friction (δ) of the powder.

When $d_p/R_w < 1$, μ_d is influenced by both the powder and wall properties. For cases where the powder particles are softer than the wall ($H_p/H_w < 1$), shearing would result in a continuous decrease in the value of μ_d with increasing value of d_p/R_w. High particle stresses at the wall may cause fracture of particles of brittle material, leading to the formation of a 'sticking' layer and hence higher values of μ_d.

As the value of d_p/R_w increases, the total stress in a powder particle decreases due to the increasing number of contact points with the wall. Thus, increasing the particle size or having a smoother wall would lessen the chance of particle fracture. Friction measurements of such powders demonstrate a gradual transition of mainly powder-powder friction to powder-wall friction with increasing values of d_p/R_w.

The present study involves powders with par-

TABLE 2

Angle of wall friction (Φ) and angle of effective friction (δ)

	Starch 1500			Avicel PH101			Calcium carbonate			Maize starch		Lactose B		
Code:	S1	S2	S3	A1	A2	A3	C1	C2	C3	M1	M2	L1	L2	L3
Size fraction (μm)	<11	11–23	23–45	<11	11–23	23–45	<11	11–23	23–45	<11	11–23	< 5	5–11	11–19
Plate St														
tan Φ	0.228	0.164	0.174	0.136	0.104	0.110	0.189	0.136	0.132	0.166	0.125	0.483	0.405	0.334
Φ (°)	12.8	9.3	9.9	7.7	5.9	6.3	10.7	7.7	7.5	9.4	7.1	25.8	22.0	18.7
Plate Mt														
tan Φ	0.222	0.281	0.236	0.280	0.209	0.189	0.546	0.471	0.402	0.250	0.202	0.633	0.708	0.695
Φ (°)	12.5	15.7	13.3	15.6	11.8	10.7	28.6	25.2	21.9	14.0	11.4	32.3	35.3	34.8
tan δ	0.805	0.758	0.674	0.819	0.846	0.808	0.632	0.644	0.608	0.622	0.707	0.823	0.825	0.84
δ (°)	38.8	37.2	34.0	39.3	40.2	38.9	32.3	32.8	31.3	31.9	35.5	39.5	39.5	40.0

St, smooth textured; Mt, medium textured.

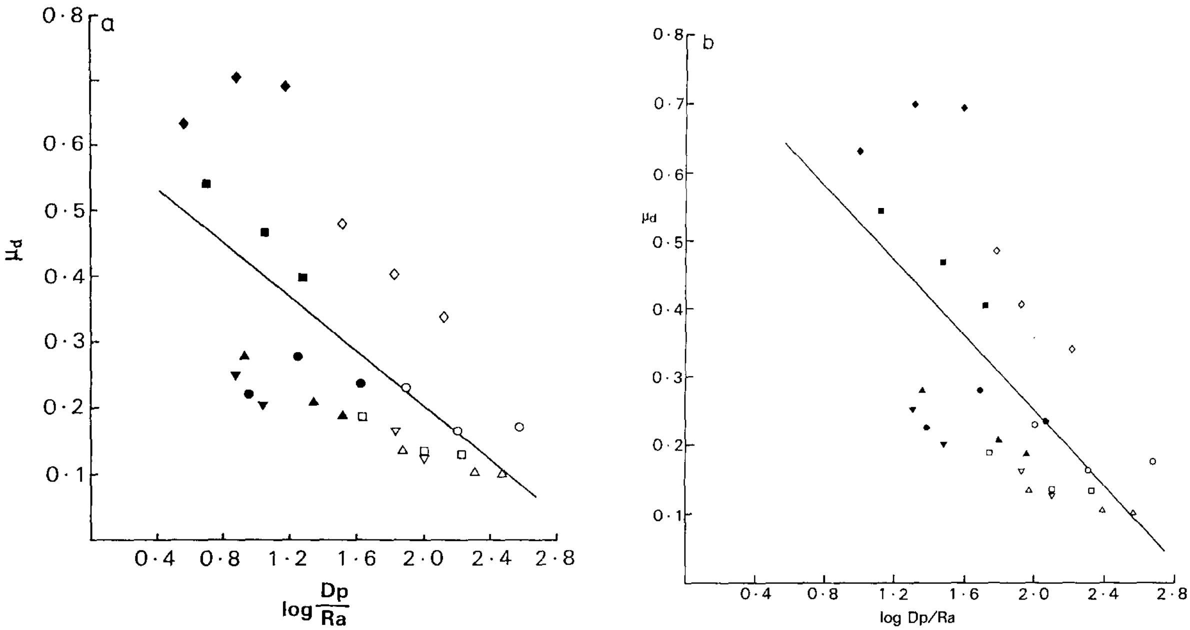

Fig. 4. Coefficient of fraction (μ_d) as a function of $\log(D_p/R_a)$. D_p, mean particle size; R_a, values measured (a) radially or (b) circumferentially along the plate. Correlation coefficient, $r = -0.654$; $n = 28$. Key to symbols:

Powder code	Annular plate	
	St	Mt
S	○	●
A	△	▲
C	□	■
M	▽	▼
L	◇	◆

ticle size greater than R_w ($= R_a$) and the hardness of the particles is assumed to be less than that of the wall. The evidence from Fig. 4a and b generally seems to support the postulate of Strijbos (1976, 1977). For each powder system, $\tan \Phi$ ($= \mu_d$) tends to decrease with increasing values of D_p/R_a as discussed earlier. The fact that lactose and calcium carbonate size fractions show higher values of $\tan \Phi$ as compared to other powders suggests that these powders have a greater tendency to fracture during experimentation, hence contributing to a higher degree of powder-powder friction. Examination of the plate surfaces after shearing confirms a greater adherence of particles of these two powders on the surfaces, especially on the rougher textured plate (Mt). In fact, for lactose and calcium carbonate powders, the values of Φ obtained tend to approach their angles of effective friction, δ (Table 2). The lower values of Φ, seen for the smallest size fraction of lactose (L1) and Starch 1500 (S1) on the rougher textured plate (Mt), may be due to greater variations in the area and extent of powder build-up on subsequent tests.

Other powder systems show a linear relationship between wall friction and particle size (i.e. the value of Φ decreases as particle size increases).

Conclusions

The angles of wall friction (Φ) and powder-wall adhesion are functions of the powder material, its particle size and the wall texture. For a particular excipient, smaller values of Φ are generally exhibited by larger size fractions and a smoother textured wall.

Acknowledgment

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