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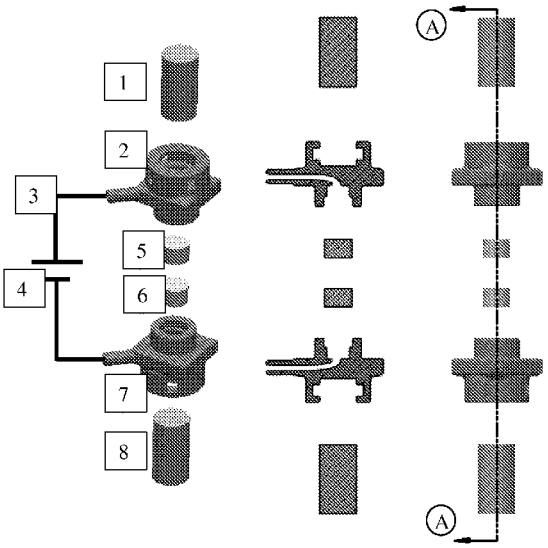
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Figure 1

Fig.1a) Fig.1b) Fig.1c)




(57) Abstract: The present invention relates to a device designed to adapt to a Capillary Breakup Extensional Rheometer (CaBER) or to a Filament Stretching Extensional Rheometer (FiSER). The adapter (A) comprises two insulated plastic components, which are connected to upper and lower shafts of the rheometer device. Stainless-steel plates are inserted inside the plastic components and connected to the DC voltage source via copper wires. By connecting the adapter (A) to a CaBER or FiSER-device it is possible to apply an electric field to the sample fluid aligned with the extensional flow exerted by said rheometer devices for the characterization of the rheological properties of electrorheological fluids (ERFs) under extensional flow of rheometers. This invention is thus in the technical field of electrorheological fluids (ERFs) with rheological properties and is useful for measuring and characterizing the properties of such fluids.

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# DESCRIPTION

# EXTENSIONAL ELECTRO-RHEOLOGICAL ADAPTER

## Technical field

The present invention relates to a device designed to adapt to a Capillary Breakup Extensional Rheometer (CaBER) or to a Filament Stretching Extensional Rheometer (FiSER).

The adapter (A) comprises two insulated plastic components, which are connected to upper and lower shafts of the rheometer device. Stainless-steel plates are inserted inside the plastic components and connected to the DC voltage source via copper wires.

By connecting the adapter (A) to a CaBER or FiSER-device it is possible to apply an electric field to the sample fluid aligned with the extensional flow exerted by said rheometer devices for the characterization of the rheological properties of electrorheological fluids (ERFs) under extensional flow of rheometers.

This invention is thus in the technical field of electrorheological fluids (ERFs) with rheological properties and is useful for measuring and characterizing the properties of such fluids.

## Background of the invention

Electrorheological fluids (ERFs) are suspensions of extremely fine and electrically active particles in an electrically insulating fluid. Under the presence of an external electric field, their rheological properties (viscosity, yield stress, shear modulus, etc.) change reversibly with response time on the order of milliseconds.

Under the electric field, ERF can suddenly increase its viscosity and even change from liquid-like state to solid-like state accomplished by yield stress to resist shearing deformation. This phenomenon is called the electrorheological effect. ERFs have attracted considerable interest in recent decades due to their wide potential uses in active control of conventional and intelligent devices. These fluids are typically used in fast-acting clutches, hydraulic valves, brakes, shock absorbers, tactile displays and flexible electronics.

Several comprehensive reviews have been published on ERFs and the mechanism of the electrorheological effect. A generally accepted description for the electrorheological effect is electric polarization mechanism. Let us consider a suspension of particles in a fluid medium. The particles and the fluid are assumed to be electrically nonconducting or only slightly conducting.

Due to the difference in the dielectric constant of the fluid and solid, the particles will be electrically polarized when an electric field is applied to this colloidal suspension. Under this condition, the random dispersion is not the lowest energy state of the system according to the resulting induced

dipole-dipole interaction between the particles. Consequently, the particles tend to aggregate and form chains along the direction of the applied electric field. The formation of the chains is the reason why ERFs exhibit an increased viscosity and even solid-like behaviour in the presence of an electric field.

For a complete description and better understanding of the performance of ERFs under real applications, the rheological properties of them must be measured in various canonical flows.

For simple shearing flows many rotational rheometers are conveniently modified with appropriate devices to apply an external electrical field to the sample, while it undergoes the shear deformation. However, measurements of rheological material functions in uniaxial extensional flows are still relatively new, especially for low viscosity solutions. In this respect, in the absence of an electrical field, ERFs are fairly mobile systems and data under the uniaxial extensional flow conditions is simply non-existing.

Since the pioneering works filament stretching rheometry has attracted much interest among the various methods developed for characterization of extensional flows. A major advantage of this method is that the velocity field far from the rigid end plates is essentially one- dimensional and purely extensional.

In consequence, filament stretching rheometry is considered an accurate technique to characterize the extensional response of mobile liquids.

In recent decades, a plethora of filament stretching configurations for generating extensional flows have been proposed.

Two of these configurations have shown enhanced performance and simplicity for rheometric purposes:

- A capillary breakup extensional rheometer (CaBER™) imposes an extensional step strain of order unity and the filament subsequently thins under the effect of capillary forces without additional kinematic input at the boundaries. As a result, the fluid filament undergoes a thinning process with an extensional strain rate defined by the interfacial tension and extensional properties of the fluid. Large extensional strains can still be attained at the mid-region as it progressively necks down and eventually breaks. Typically, the time evolution of the midpoint diameter of the thinning filament is the only measured quantity, and the exponential decay of the filament diameter with time determines the relaxation time of the viscoelastic liquid.
- The filament stretching extensional rheometer (FiSER™) imposes an exponential velocity to the upper plate in order to create a uniaxial extensional flow with constant strain rate. In this rheometer, the transient extensional viscosity is calculated based on the measurement of the temporal evolution of the tensile force exerted by the fluid column on the bottom stationary end plate and the filament radius at the axial midpoint of the filament.

Although FiSER technique is suitable for characterization of viscous polymer solutions and melts, its use can be precluded with low viscosity solutions due to the force transducer accuracy, and gravitational and inertial effects.

In this case, using the CaBER for filament breakup investigations is a cheaper alternative for measuring extensional properties of the fluid.

Important information about the relaxation time, breakup time and the non-Newtonian behaviour of the fluid can be achieved by monitoring the filament thinning process after a rapid extensional deformation. Because of all these reasons, capillary breakup rheometry is being used more frequently for characterization of extensional properties of complex fluids, such as viscoelastic fluids, shear thickening fluids, or yield stress fluids.

While both devices (FiSER and CaBER) can characterize the extensional behaviour of a wide range of complex fluids, neither of them supplies an external electric field while executing the external deformation. Also, neither of them has currently the capability of characterizing the extensional properties of ERFs under the action of the electric field.

Therefore, there is a need of developing a tool that allows the characterization of the rheological properties of a wide range of ERFs in CaBER- or FiSER- rheometer devices.

The present invention provides such tool as an adapter to said rheometer devices allowing the application of an external electric field aligned with the flow kinematics undergone by the fluid sample while the extensional characterization is taking place.

## Description of the figures

Figure 1 represents one embodiment of the adapter of the invention, wherein:

**Fig.1a** represents a 3-D view of the adapter (A),

**Fig.1b** represents a cross-sectional view A-A of the adapter (A), and

**Fig.1c** represents the front view of the adapter (A).

The following references numbers represent:

1. Upper shaft of a CaBER device,
2. Upper plastic component of the adapter (A),
3. A cooper wire,
4. A DC voltage source,
5. Upper stainless-steel plate of the adapter (A),
6. Lower stainless-steel plate of the adapter (A),
7. Lower plastic component of the adapter (A), and
8. Lower shaft of a CaBER device.

**Figure 2** represents an assembly of the adapter (A) with a CaBER device for electrorheological measurements, wherein:

9. High speed camera,
10. Light source,
11. Laser system.

The adapter (A) is connected to the shafts (1,8) of the CaBER device, and the copper wires (3) connect the plates (5,6) of the adapter (A) the power source (4).

In this representation, it is possible to observe that the assembly with the adapter (A) does not block the view of the laser (11) nor of the camera (9).

**Figure 3** represents the time evolution of the filament thinning process for an electrorheological sample with 30 wt% concentration at different applied voltages of 1kV and 2kV. It can be observed that, under extensional flow and no electric field applied, the sample behaves as a Newtonian fluid, showing a linear relationship between time and $d/D_{plate}$[-].

## Description of the invention

The present invention relates to device (A) designed to adapt to a Capillary Breakup Extensional Rheometer (CaBER) or to a Filament Stretching Extensional Rheometer (FiSER).

Rheological properties of any fluid and in particular of electrorheological fluids must be measured in various canonical flows for a complete description and for the better understanding of their performance under real applications as they mostly involve highly complex flows.

For simple shearing flows many rotational rheometers can be conveniently modified with appropriate devices to apply external electrical fields to the sample, while it undergoes the shear deformation.

However, measurements of rheological material functions in uniaxial extensional flows are still relatively new, especially for low viscosity solutions.

In this respect, in the presence of a mild electrical field, electrorheological fluids are fairly mobile systems and data under the uniaxial extensional flow conditions is simply non-existing.

The adapter (A) comprises two insulated plastic components (2,7), which are connected to upper and lower shafts (1,8) of the CaBER. Stainless-steel plates (5,6) are inserted inside the plastic components (2,7) and connected to the DC voltage source (4) via copper wires (3). The copper wires (3) further connect plates (5,6) to the power source (4).

Therefore, in an embodiment of the present invention the adapter (A) comprises

- two insulated plastic elements (2,7) connectable to the upper shaft (1) and to the lower shaft (8) of said rheometer devices,
- two stainless-steel plates (5,6), which are inserted inside two correspondent plastic components (2,7), and
- copper wires (3) able to connect the plates (5,6) and plastic elements (2,7) to an external power source (4).

The adapter (A) can be provided in different sizes in order to match with the features of the rheometer devices to be attached to. For that purpose, the stainless-steel plates (5,6) can be provided in different diameter sizes, such as of 4mm, 6mm, 8mm, depending on the rheometer CaBER or FiSER device.

By connecting the adapter (A) to a CaBER or FiSER-device it is possible to apply an electric field to the sample fluid aligned with the extensional flow exerted by said rheometer devices for the characterization of the rheological properties of electrorheological fluids (ERFs) under extensional flow of rheometers.

In another embodiment, the adapter of the invention further comprises an external power source (4) capable of providing the adapter (A) with an electrical voltage of $\geq$ 0kV to $\leq$ 3.

The behaviour of an ERF under extensional flow with and without the application of electric field and the results can be characterized in a reproducible way. Further, it is also very interesting from the scientific and technological points of view.

After accessing filament thinning process of the electrorheological sample without applying voltage and when voltages of 1 and 2 kV are applied, it is possible to observe that under extensional flow and no electric field applied, the sample behaves as a Newtonian fluid, showing a linear relationship between time and $d/D_{plate}[-]$.

When the external electric field voltage increases to 1kV, the filament thinning process delays when compared to the previous case in which no field was applied.
This is a clear evidence of the presence of a microstructure formed in the ERF sample particles under the presence of the field. That microstructure is responsible for a viscoelastic behaviour.

When the external electric field is further increased to 2kV then it is possible to observe that the filament does not break.

This is because the internal microstructure is now so developed that bridges the distance between plates and surface tension is not strong enough to cut it out. This observation confirms that the adapter (A) of the invention provides an efficient electric field to the sample.

## Examples

### Example 1. Characterization of the Adapter (A)

Adapters (A) was produced in different sizes (A1, A2 and A3). All adapters (A) comprise two insulated plastic components (2,7), which are connected to upper and lower shafts (1,8) of the CaBER, 2 stainless-steel plates (5,6), which are inserted inside correspondent plastic components (2,7), which are connected to the DC voltage source (4) via copper wires (3). The copper wires (3) further connect plates (5,6) to the power source (4), as shown in Fig.1.

The plate diameters are of 4mm for A1, 6mm for A2 and 8mm for A3, matching the size of the plate in CaBER device.

In this way, it is possible to have the choice of measuring the time evolution of the filament diameter either by the laser micrometer available (11) in the CaBER, by using a high-speed camera (9), or both simultaneously, as shown in Fig.2.

### Example 2. Characterization of the behaviour of an ERF

The behaviour of an ERF was characterized under extensional flow with and without the application of electric field.

The ERF was prepared by producing a suspension of corn-starch in olive oil with a concentration of 30 wt%.

Fresh samples are used for each measurement after being redispersed in an ultrasound bath (Velleman VTUSC3) for 300s

since the rheological properties of the suspension may depend on the dispersion quality.

Then, images were recorded by using a Matlab device.

**Figure 3** shows the filament thinning process of the electrorheological sample without applying voltage and when voltages of 1 and 2 kV are applied.

In this case, the 4 mm diameter plates are selected and the initial gap between the plates is fixed at 2 mm.

The final axial separation between the plates of 6 mm is fixed and the initial stretch profile is linear with a "strike time" of 20 ms.

It can be observed that, under extensional flow and no electric field applied, the sample behaves as a Newtonian fluid, showing a linear relationship between time and $d/D_{plate}[-]$.

When an external electric field is 1kV, the filament thinning process delays compared to the previous case in which no field was applied. This is a clear evidence of the presence of a microstructure formed by the corn-starch particles under the presence of the field.

That microstructure is responsible for a viscoelastic behaviour. If the external electric field is increased further up to 2kV, then it is observed that the filament does not break. This is because the internal microstructure is now so developed that bridges the distance between plates and surface tension is not strong enough to cut it out. This observation

confirms that the devices supply the electric field efficiently to the sample.

## CLAIMS

1. An adapter device (A) for connecting to Capillary Breakup Extensional Rheometer (CaBER) or to a Filament Stretching Extensional Rheometer (FiSER) device **characterized for** comprising:
   - two insulated plastic elements (2,7) connectable to the upper shaft (1) and to the lower shaft (8) of said rheometer devices,
   - two stainless-steel plates (5,6), which are inserted inside two correspondent plastic components (2,7), and
   - copper wires (3) able to connect the plates (5,6) and plastic elements (2,7) to an external power source (4).

2. An adapter device (A) according to claim 1 **characterized by** the stainless-steel plates (5,6) are provided in three different sizes of 4mm (A1), 6mm (A2) and of 8mm (A3).

3. An adapter device (A) according to any of the claims 1 or 2 **characterized by** further comprising an external power source (4) capable of providing the adapter (A) with an electrical voltage of $\geq$ 0kV to $\leq$ 3kV.

Figure 1

Fig.1a) Fig.1b) Fig.1c)

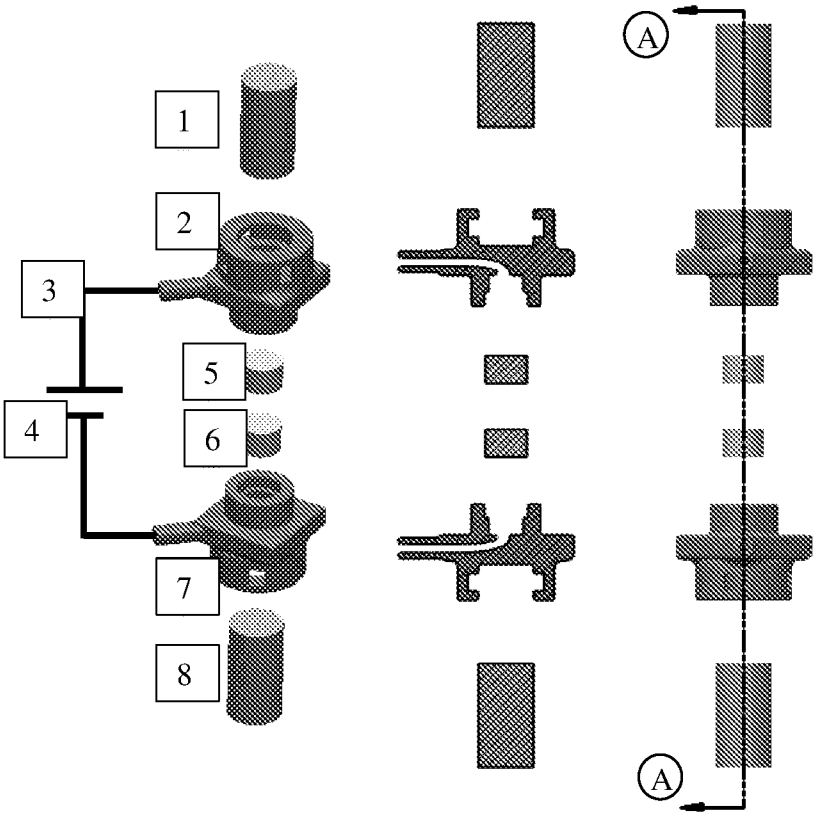


Figure 2

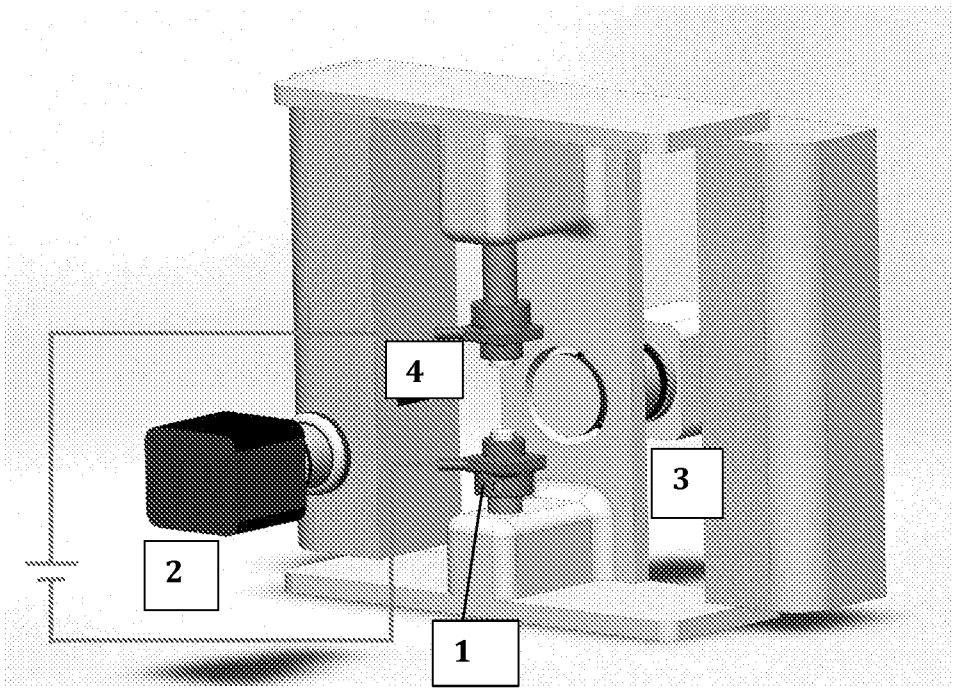
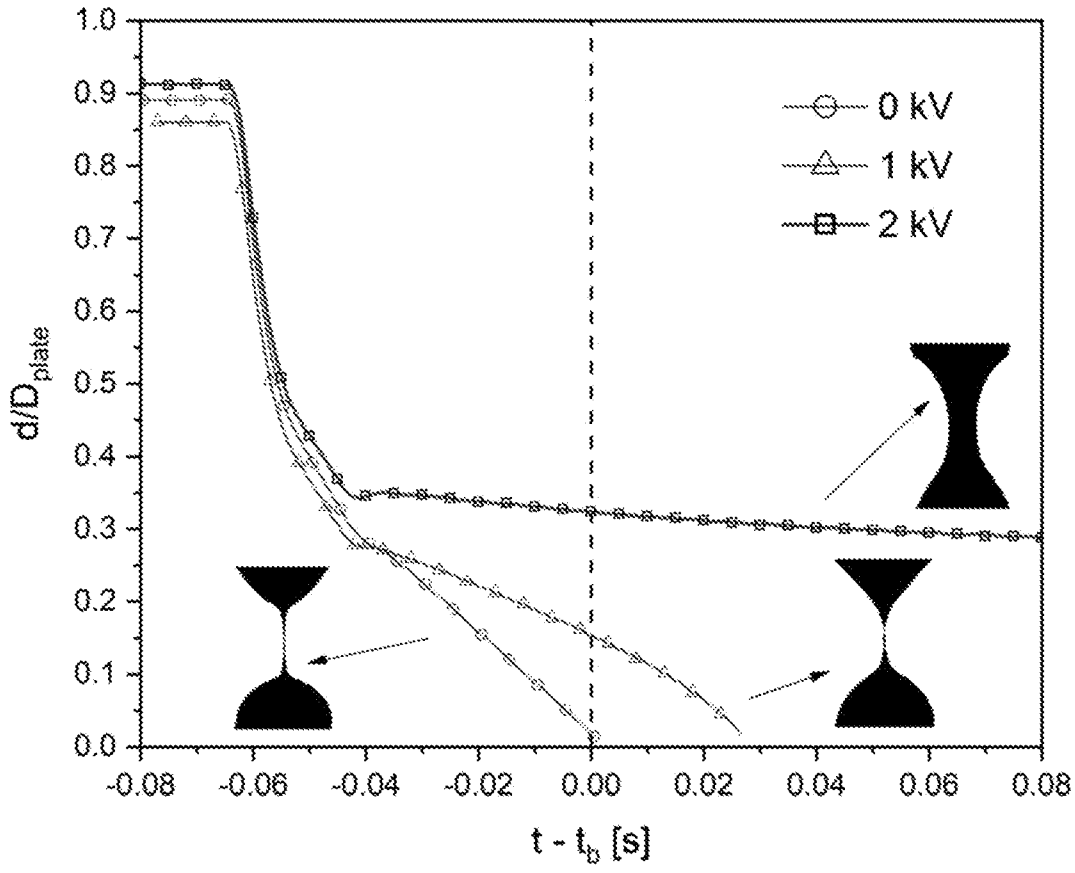



Figure 3



# INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. G01N3/06 G01N3/08 G01N11/00 G01N13/02
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X | CN 101 089 591 B (CHINESE ACAD PHYSICS INST) 26 January 2011 (2011-01-26) paragraph [0024] - paragraph [0027] figure 1 | 1-3 |
| A | CN 100 381 808 C (UNIV SHANGHAI JIAOTONG [CN]) 16 April 2008 (2008-04-16) the whole document | 1-3 |
| A | Ta Instruments: "ARES-G2 RHEOMETER", , 1 January 2018 (2018-01-01), XP055719396, Retrieved from the Internet: URL:https://www.tainstruments.com/wp-content/uploads/BROCH-ARESG2-2014-EN.pdf [retrieved on 2020-07-31] page 30 | 1-3 |

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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| Date of the actual completion of the international search | Date of mailing of the international search report |
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| 31 July 2020 | 13/08/2020 |

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| European Patent Office, P.B. 5818 Patentlaan 2<br>NL - 2280 HV Rijswijk<br>Tel. (+31-70) 340-2040,<br>Fax: (+31-70) 340-3016 | Reto, Davide |

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IB2020/053362

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---|---|---|
| CN 101089591 B | 26-01-2011 | NONE | |
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