Research Review on Devices and Methods for Rapid Measurement of Paper Ash

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The Chinese national standard for paper ash measurement cannot meet the requirements for accurate and rapid ash measurement in actual production and scientific research because of the long measuring time, tedious procedures, and large human error. This paper reviews some worldwide devices and methods for rapid measurement of paper ash, including ceramic fiber muffle furnace, microwave muffle furnace, the addition of ash adjuvant, dry samples method, direct combustion of paper samples, oxygen-enriched combustion method, chemical analysis method, and ray method, etc. The differences and relationships are identified among these devices and methods. By comparing the different ash measurement methods, the rapid ash analyzer based on X-ray technology has the obvious advantages of short measuring time and small error. Lastly, the present situation and the development potential of these devices and methods are discussed in this review.

Keywords: Paper; Ash; Rapid measurement; Muffle furnace; X-ray; Device; Method

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INTRODUCTION

Paper ash is an important parameter of paper quality (Huang 1990). Paper for different uses has corresponding ash range requirements. Too high or too low ash can lead to the deterioration of the paper, resulting in deterioration in production (Gu and Sun 1993). The measurement and control of paper ash has become an important part in actual production and scientific research. Accurate control can improve the stability of paper quality and reduce the fluctuation of ash content due to the change of pulp ratio, or the types and properties of paper fillers (Gu and Liu 2011). The cost of paper can be reduced by increasing the level of paper ash (Xiao 2012; Liu 2013), but the large increase of ash will affect the main characteristics of the paper, such as the thickness, internal bonding strength, folding resistance, and so on (Zhou and Fan 2008). However, according to the Chinese national measurement standard, the paper is carbonized and burned by a traditional muffle furnace in the process of measurement, which has many disadvantages such as tedious procedures, long measuring time, large temperature deviation, and an uneven temperature distribution in the furnace (Li et al. 2015). Therefore, the Chinese national measurement standard cannot meet the requirements for accurate and rapid ash measurement in actual production and scientific research. To overcome the disadvantages of the Chinese national measurement standard and meet the ash measurement requirements in actual production, the relevant scientific research institutions and paper enterprises have improved the Chinese national measurement standard and developed a series of devices and methods for the rapid measurement of paper ash. This paper introduces these devices and methods.
IMPROVEMENT OF THE CHINESE NATIONAL MEASUREMENT STANDARD

The Chinese national measurement standard puts the crucible containing the sample in the traditional muffle furnace. The temperature is set at 900 ± 25 °C, and the burning time is 1 h. After burning, the sample cools in the air for 10 min, and then is moved into the desiccator to cool to room temperature. The residue is then weighed to calculate the paper ash (Guan et al. 2008). It is worth noting that the operator must avoid scalding their hands due to the high temperature when removing the samples. However, the traditional muffle furnace has many disadvantages, such as slow heating, uneven temperature distribution, and tedious procedures. Based on the disadvantages of the Chinese national measurement standard, many scientific research institutions and paper enterprises have improved the muffle furnace device and the carbonizing and burning crafts of paper.

Improvement of the Traditional Muffle Furnace

Ceramic fiber muffle furnace

The ceramic fiber muffle furnace uses ceramic fiber as the heat insulating material, which is composed of solid filaments and pores. The ceramic fiber can hinder the exchange of heat and has good heat insulation ability. Also, it has the characteristics of rapid heating, good insulation, and electricity saving (Ke 2004). Thus, the ceramic fiber muffle furnace can overcome the disadvantages of slow heating and high energy consumption in the traditional muffle furnace, and it can greatly reduce the time of analyzing paper ash.

Microwave muffle furnace

The microwave muffle furnace is a heating furnace with a microwave energy electromagnetic field as the heating element. The microwave muffle furnace is easy to control, and the rate of heating is fast. It can be heated from room temperature to 1000 °C in a few minutes. Also, when using a microwave muffle furnace to measure paper ash, there is no need to carbonize the paper; rather, the paper ash is directly converted to ash. It is simple to operate and most samples can be completely ashed within 10 min which is nearly 100 times faster than the traditional muffle furnace. Moreover, the samples are cooled quickly after ashing. The device can be cooled to room temperature within 6 seconds, which takes about an hour or more in the traditional muffle furnace. In addition, the microwave muffle furnace is equipped with an automatic emptying system that can guarantee the operator’s safety (Jiang et al. 2012). However, the microwave muffle furnace has the disadvantages of small volume and high price (Ke 2004). He et al. (2013) used the PHOENIX microwave muffle furnace to measure the ash content of cigarette paper. The results showed that the intra-day relative standard deviation was 0.38% and when comparing results obtained on different days the relative standard deviation was 0.52%. Moreover, the results were consistent with those of the Chinese national measurement standard, and the precision was better than that of the Chinese national measurement standard.

Improvement of the Paper Burning Craft

Reduce the amount of paper sample

When measuring the paper ash by the Chinese national measurement standard, the quality of the paper sample required for measurement must meet the ash content of 2 mg or more after burning. The amount of paper sample is usually between 2 g and 3 g. Generally, the content of filler in paper is between 5% and 35%. Thus, the amount of ash
sample is about 0.5 to 1.0 g, which not only meets the requirements of the Chinese national measurement standard, but it also reduces the time of burning and ashing in the muffle furnace. Moreover, it accelerates the ashing process.

The addition of ash adjuvant

Thermal degradation of cellulose and hemicellulose occurs during the process of carbonizing and burning. The addition of H₂O₂ before paper carbonization promotes the degradation of cellulose and hemicellulose (Pan et al. 2010). Zhang (2012) found that sufficient oxygen in the paper burning process can increase the burning temperature, speed up the burning, and promote the complete combustion of the paper. Thus, it accelerated the burning and ashing process of the paper, reduced the ashing time, and improved the measurement accuracy.

Dry Sample Method

Cigarette paper ash is an important factor in cigarette paper quality. It not only can improve air permeability, adjust the burning rate, and improve the whiteness and opacity, but also it can improve the feel of cigarette paper and save paper fiber consumption (Wu and Li 2005; Yu et al. 2010; Cai 2010). The traditional measurement of cigarette paper ash has so many disadvantages that it cannot meet the demand for the actual measurement. Jing et al. (2016) used the dry sample to measure the cigarette paper ash, eliminating the need to measure the moisture content of cigarette paper and reducing intermediate links. The variance of the ash measurements was evaluated (Zhang 2003). The results showed that the repeatability of measurement was good. Moreover, there was no significant difference from the results of the Chinese national measurement standard, which overcame the disadvantages of the long measuring time and tedious procedures. The improved method can be used as the method of measuring cigarette paper ash.

Direct Combustion of Paper Samples

Yin and Li (1990) made the paper samples into paper strips with a length of 10 cm and a width of 3 cm. They put these paper strips on the wire in a certain gap and obtained the ash data according to the change in quality before and after burning. Then they fitted and regressed the measured ash content data with the standard ash content data, and calculated the ash content through the regression formula. The results showed that the relative error between the calculated ash content and the standard ash content was less than 3.30%. The more rapid method was able to effectively guide workshop production and enable prompt prediction of the paper ash value. Gu and Liu (2011) used the Chinese national measurement standard and the rapid measurement method to measure the ash content of paper samples filled with talcum powder and heavy calcium. Besides, they obtained the coefficient between the two by comparing and analyzing the ash content data, further optimized the rapid measurement method, improved the ash content qualified rate in the workshop, reduced the slurry consumption, and ensured the product quality. Li et al. (1996) used the carbonization coefficient method to rapidly measure the ash of newsprint. This method carbonized and burned the paper samples in a special rapid measuring device to calculate the ratio of carbonization to the ignition weight of the paper samples, which was named “carbonization coefficient”. Moreover, this method also calculated the arithmetic mean of the carbonization coefficient of the randomly selected batch paper samples. According to the principle that the carbonization coefficient actually produced in the same period was relatively stable, the ash content of the paper sample can be directly
measured by the carbonization of the sample. Thus, the number of operational steps was greatly reduced, thereby realizing the rapid measurement of the newsprint ash and reducing the time from 8 hours to 10 minutes. Although the method can quickly measure newsprint ash, the paper sample was selected in a small amount during the measurement of the carbonization coefficient. Also, the paper sample cannot be completely burned. Even worse, the combustion caused partial decomposition of the filler, resulting in low accuracy and poor stability of the measurement data.

Since paper is a porous material and has good thermal insulation and penetrability (Mendes et al. 2001), the paper sample will undergo a smoldering process after the combustion flame disappears, which further leads to the loss of the filler in the paper sample, affecting the accuracy of the ash measurement. Therefore, before the ash measurement, the paper sample should be cut into strips or the paper sample should be burned with enough oxygen to make it burn completely.

**Oxygen-enriched Combustion Method**

The oxygen-enriched combustion method is a combustion method that uses oxygen-enriched air with high oxygen content to support combustion (Liu 2007; Dai et al. 2000; Li and Wang 2003). In the process of oxygen-enriched combustion, the increase of oxygen content greatly increases the flame temperature, reduces the ignition temperature and burnout temperature of the fuel, and improves the complete combustion efficiency of the fuel (Jiao et al. 2010). Based on the above principles and characteristics, the German company Grenier und Grabner GmbH developed an instrument for rapidly measuring paper ash, which is shown in Fig. 1.

![Fig. 1. Instrument for rapidly measuring paper ash (reprinted with permissions from German Grenier und Grabner GmbH Co.)](image)

The device puts the paper sample into a metal mesh in the combustion chamber for combustion and performs rapid measurement of the ash by controlling the amount of oxygen. The measuring time is about 5 min. Moreover, the combustion chamber and metal mesh can effectively reduce the volatilization of solid components in the paper combustion process. The metal mesh can be cooled to room temperature in a short time, thereby shortening the measuring time of paper ash. However, this instrument had certain dangers when igniting the paper sample. For example, the combustion chamber contains a certain amount of oxygen, it may burn hands when igniting the paper sample. Thus, safety precautions such as wearing special gloves should be taken during operation.
CHEMICAL ANALYSIS METHOD

The Chinese national measurement standard and related improvements need to carbonize or burn the paper when measuring the paper ash, resulting in the loss of the corresponding fillers and affecting the measurement accuracy of the ash. To solve the disadvantages in the Chinese national measurement standard, the relevant scientific research institutions have developed the chemical analysis method to rapidly measure the paper ash.

**Ion Chromatography Method**

Calcium carbonate is divided into light calcium carbonate (PCC) and heavy calcium carbonate (GCC), where the former is used to produce paper and the latter is used as coating pigment (Liang 2009; Zhao et al. 2009). Moreover, light calcium carbonate not only can increase the refractive index of cigarette paper, but also it can increase the whiteness and air permeability of cigarette paper. At the same time, its dosage is an important factor in cigarette paper performance (Wang et al. 2002; Zhang et al. 2006; Wang 2000, 2007). To accurately measure the calcium carbonate content in cigarette paper, understand the stability of process parameters, and evaluate product quality (Liu and Guan 2001; Cui 2003), Lu et al. (2011) used ion chromatography method to measure the calcium ion content in cigarette paper and calculate calcium carbonate content. In this method, 10 cigarette paper samples were respectively added to 100 mL of 1% acetic acid aqueous solution, ultrasonically extracted at room temperature for 40 min. Besides, the calcium ions in cigarette paper samples were measured using an ICS-3000 ion chromatograph (DIONEX USA) and calculated according to the relative molecular mass relationship. As a result, the measurement limit was 0.007 mg/g, the recovery rate was 95.20% to 99.50%, and the precision was 0.60%, which effectively helped the technician to monitor the product quality. Moreover, the analysis rate was fast, and this method was suitable for large-scale sample measurement. However, the promotion of this method was affected by the use of many instruments and high prices.

**Indicator Titration Method**

As a kind of paper filler with low price, good quality and easy access, calcium carbonate is widely used by paper enterprises. Its content plays an important role in the performance of paper filled with calcium carbonate (Zhang and Wang 2004). In addition to the Chinese national measurement standard, Zeng (2001) developed a phenolphthalein indicator titration method to rapidly measure paper ash based on the principle that calcium carbonate can completely react with hydrochloric acid. In this method, 2 g paper samples were cut into pieces with a width and length of 1 cm each. The pieces were placed in an Erlenmeyer flask with distilled water and hydrochloric acid, and they were boiled for 1 minute. After cooling, the phenolphthalein indicator was added into the Erlenmeyer flask, and the calcium carbonate content in the paper sample was measured according to the volume of sodium hydroxide consumed at the end of the titration. The phenolphthalein indicator titration method avoided the element loss caused by the Chinese national measurement standard, and it had good data stability and short operation time. Moreover, it can be applied to measure paper ash in actual production. However, the ash measured by this method can only be a part of the ash that reacted with hydrochloric acid. Thus, the measurement result was lower than the true ash content. Even worse, there was a problem.
that the titration endpoint was difficult to distinguish when measuring colored paper or a turbid liquid (Zheng 1991).

In order to study the differences in the composition and content of inorganic additives in domestic and imported reconstituted tobacco leaves, Wang et al. (2015) used the potassium permanganate method to measure the content of artificially added calcium carbonate in the reconstituted tobacco leaves. This method firstly separated some plant calcium in the sample. Then they extracted artificially added calcium carbonate in the sample and converted it into calcium oxalate precipitate. Finally, they used the dilute sulfuric acid to dissolve the precipitation, added potassium permanganate standard solution, and calculated the content of calcium carbonate in the sample according to the volume of potassium permanganate solution consumed at the end of titration. Although this method can rapidly measure the content of artificially added calcium carbonate in the reconstituted tobacco leaves, some plant calcium was mixed into the artificially added calcium carbonate in the process of extraction and separation. Thus, the measurement result was larger than the actual measurement result. Even worse, it was difficult to distinguish the endpoint of the titration in the case of measuring colored paper or turbid liquid.

**Potentiometric Titration Method**

To compensate for the disadvantages of the indicator titration method and meet the needs of production quality control, Wei and Yang (1991) studied a potentiometric titration method for the measurement of calcium carbonate in the cigarette paper. In this method, 2 g paper samples were cut into pieces with a width and length of 5 mm each, which were placed in a beaker and boiled with sulfuric acid for 2 to 3 min. After cooling, sodium hydroxide solution was added into the beaker for the potentiometric titration, and the endpoint was distinguished by use of a digital display type pH meter. Then, the calcium carbonate content in the paper would be calculated according to the volume of sodium hydroxide consumed at the end of the titration. This method used an instrument instead of an indicator to distinguish the endpoint of the titration, reducing the measurement error. Besides, it was also suitable for the measurement of colored paper or turbid liquid and had the advantages of wide applicability, small measurement error, and simple operation. Peng et al. (2008) developed a method for measuring the cigarette paper ash by the pH/ISE (Ion Selective Electrode) tester and automatic potentiometric titrator. The results showed that the recovery rates of the pH/ISE tester and automatic potentiometric titrator were respectively 100.00% to 100.40% and 99.80% to 100.80%. The maximum variance was 0.62% to 0.96%. Moreover, when the confidence interval was 95.00%, there was no significant difference in the precision and deviation between the potentiometric titration and the indicator titration.

**Complexometric Titration Method**

Both the indicator titration method and the potentiometric titration method measured the paper ash based on the acid-base titration principle. However, the paper contained other acid-soluble metal oxides and carbonates in addition to calcium carbonate (Pan et al. 2006; Hou et al. 2007; Liu et al. 2007). This caused errors in the ash measurement results. In order to solve the disadvantages of indicator titration method and potentiometric titration method, Zhou et al. (2012) used the complexometric titration method to measure the paper ash. The complexometric titration method firstly dissolved the calcium carbonate in the cigarette paper. After that, the appropriate amount of triethanolamine that can block the metal ions such as Mg$^{2+}$ was added dropwise. Then, the
disodium EDTA (Ethylene Diamine Tetraacetic Acid) was used as the titration solution, and the calcium indicator was selected under the condition of pH 12.0 to distinguish the endpoint of the titration. Since the triethanolamine masked the interference of high-valent metal ions, the measurement results had higher accuracy and this method had a better applicability.

RAY METHOD

The titration method and complexometric method are suitable only in cases where calcium carbonate represents nearly 100% of the paper fillers. Moreover, the chemical analysis method is a destructive measurement method that cannot be considered for the role of an industrial online measurement. To solve the disadvantages in the chemical analysis method, the relevant scientific research institutions have developed the ray method to rapidly measure the paper ash. But it is worth noting that safety precautions such as wearing goggles and radiation protective clothing should be taken when using the ray method to measure paper ash.

Online Measurement of Paper Ash

In order to achieve online measurement and control of paper ash, reduce pulp consumption, and improve paper quality and economic benefits (Haglund and Alsholm 1980; Mo 1982; Jack 1990), Huang (1990) proposed the measurement of paper ash by fluorescent X-ray method based on the principle of selective absorption of characteristic fluorescence X-ray. Moreover, she measured 14 samples with different thickness and ash content, provided by four paper mills in Beijing. The results showed that the measuring time was 30 seconds. When paper basis weight was in the range of 28 to 120 g/m², the ash measurement error was less than 1.00% and the root mean square error was ±0.58%. However, this method was not suitable for crepe paper and capacitance paper with a basis weight of more than 200 g/m². Gu and Sun (1993) developed a soft X-ray transmission intensity measurement method. According to the characteristics that the intensity of X-ray decreases when it penetrates the material and the attenuation conforms to Beer’s law (Newman and LeDrew 2001), this method selected appropriate soft X-ray sources and detectors, and used corresponding electronic signal processing system for online measurement of paper ash. This method was simple and easy to operate. Moreover, it was also an effective measurement of ash content. Based on the principle and device of the above methods, Guo et al. (1996) used the absorption characteristics of γ-ray to study the online detector of paper ash. This detector had an ash measurement range of 1% to 30%, a measurement accuracy of ±0.50%, a suitable paper basis weight range of 20 to 150 g/m² and a reaction time of less than 50 ms. On account of the relationship between the absorption coefficient of soft X-ray and the cube of the atomic number of the substance, Ou (1998) have studied the paper ash meter, using the isotope 55Fe as the X-ray source. Moreover, they measured 28 different types of papers filled with calcium carbonate and talc. The results showed that 26 of the 28 measurement data had an error of less than 0.5%, which fully met the production requirements. Feng and Sun (1998) designed a detector for measuring paper ash online with isotopes 55Fe as the gamma-ray source based on the selective absorption characteristics of low energy X-ray. The on-site operation showed that the measurement error was less than 1%, which met the requirements for online measurement of paper ash.
Honeywell designed an ash sensor (Chen 2011) with an X-ray tube as the X-ray source based on the characteristics of paper ash preferentially absorbing X-ray (Yao and Yan 2003). The sensor can correct the ash measurement value according to the actual situation and standardize the sensor inspection to ensure the measurement accuracy of the sensor. Moreover, the sensor was equipped with an X-ray source shutter to ensure personal safety.

**Compensation and Correction of Online Measurement of Paper Ash**

The measurement of paper ash is indirect, and the paper basis weight and moisture are important factors of ash measurement. The above online measurement did not compensate and correct the paper basis weight and moisture. Therefore, according to the traditional correction methods, Feng and Sun (1997) developed a method for online compensation and correction of quantitative moisture based on a sliding average. This method performed appropriate digital filtering and correction on the sampling signal of the quantometer and moisture meter. The corrected actual value was substituted into the ash calculation formula to obtain the ash. As a result, the actual application data showed that the measurement error was less than 1.00% and the error of 25 measurement data was less than 0.60%. However, when the paper basis weight deviation exceeded ±5 g/m², the compensation and correction of this method was limited. Even worse, the error of the quantometer and moisture meter itself affected the precision of compensation and correction. Xiao (2012) used the ash sensor produced by Impact to perform ash measurement on a paper mill. The measurement error was less than 1.00%. After analyzing the disadvantages of the traditional basis weight and ash content sensors, and considering the correlation between basis weight, moisture and ash content, Shen et al. (1996) proposed a decoupling measurement method and a new type of sensor was designed and produced. This method realized the decoupling of measurement of basis weight and ash content, which made a better accuracy of the measurement data. But the selected samples were few and there was a certain contingency. By comparing the measurement data of the traditional sensor and new sensor, the new sensor was precise and excellent in performance. Moreover, the new sensor also can be applied to other fields, such as aluminum foil and compound material, etc.

**Measurement of Ash Component**

When using the online measurement method to measure paper ash, only the total ash can be determined, and ash components cannot be qualitatively and quantitatively analyzed. Bluhm et al. (1985) used the powder X-ray diffraction to quantitatively analyze the inorganic filler in paper. However, the error was so large that this method cannot meet the requirements of ash measurement. Hong et al. (1992) proposed a method for measuring ash components by X-ray diffraction technique. This method used D/max-3B X-ray to diffract the paper sample added with NaCl. Moreover, this method analyzed the diffraction angle in the diffraction pattern by Bragg's law and K value method to accurately calculate the type and content of ash component. The experimental results showed that the ash deviation was less than 0.50%, which was within the error tolerance. Rožić et al. (2004) used EDXRF (Energy Dispersive X-Ray Fluorescence) spectroscopy to perform elemental analysis on office papers of different quantitative and manufacturers. The EDXRF spectroscopy firstly pressed the paper ash into a 2 cm diameter pellet to prepare a thick target for XRF analysis. Then the Cd source was used to excite the thick target, and the amplifier circuit was used to process the current pulse generated by the X-ray and Si (Li) detector materials. Finally, the obtained spectra were analyzed qualitatively and
quantitatively by WinAxil software. The results showed that EDXRF spectroscopy can accurately measure the concentration of various elements in office paper, which greatly helped to measure the nature of office paper and research the possibility of recycling office paper. On the basis of GB/T 2679.11-2008, Li et al. (2015) proposed a method for sample pretreatment combined with infrared spectroscopy to identify a small amount of barium sulfate in paper. The method performed pickling treatment on the paper sample after ashing to remove interference of organic matter and calcium carbonate. Then it measured the barium sulfate in the paper sample by infrared spectroscopy. The results showed that the error was small and the sensitivity of infrared spectroscopy was improved. However, this method was only suitable for the qualitative analysis of a small amount of barium sulfate in paper, and there is only limited need for such information.

In response to market demand, the German Emtec company has produced a fast ash analyzer, as shown in Fig. 2. The ash analyzer can be used to measure the filler content of paper filled with CaCO₃ and CaSiO₃. Moreover, the measuring time is short and the measurement precision is high. In addition, it is especially suitable for the measurement of paper ash in the laboratory and also meets the measurement requirements of some paper mills. However, the device is expensive. And there is no similar product in the country, which provides direction and motivation for future scientific research.

In summary, based on the measuring time and error of the corresponding method, comparisons of the three methods are shown in Figs. 3 and 4. These data are quoted from the references of the corresponding methods. The samples are offset paper with a basis weight of 20 to 120 g/m². Moreover, the samples consist of plant fibers, calcium carbonate, titanium dioxide, talc and so on. It can be seen from Figs. 3 and 4 that the measuring time of the ray method and chemical analysis method are about half a minute and ten minutes respectively. And the measuring time of the Chinese national measurement standard is more than ten minutes. Moreover, the measurement error of chemical analysis method and the ray method are both about 0.50%, while the error of improvement of the Chinese national measurement standard is higher than 0.50%.

![Fig. 2. The fast ash analyzer (reprinted with permissions from German Emtec Co.)](image-url)
Fig. 3. The comparison chart of the measuring time

Fig. 4. The comparison chart of the measuring error
CONCLUSIONS

1. With the rapid development of paper enterprises, the devices and methods for rapid measurement of paper ash are developing towards improving the measurement accuracy and speed. According to the nature of paper, the relevant scientific research institutions and paper enterprises have developed a series of methods of rapidly measuring paper ash based on the Chinese national measurement standard, such as the improvement of the Chinese national measurement standard, chemical analysis method, and ray method. Moreover, these methods all meet the measure requirements of actual production.

2. Compared with the improvement of the Chinese national measurement standard, although chemical analysis method measures paper ash quickly and accurately, it cannot be used as an online measurement method. Despite the high precision and price of devices, the high environmental requirements and the small scope of application, some ray methods have short measuring time, high measurement accuracy, and low cost. Therefore, according to the needs of actual production, choosing the right ray method to measure paper ash can effectively improve production efficiency and speed up scientific research. By analyzing the advantages and disadvantages of the above methods, the X-ray diffraction method has a high cost, but the measurement speed is fast. Moreover, it not only can measure the total ash of the paper, but also it can measure the type and content of the ash component. Besides, the measurement data have good stability and high precision. It is believed that the X-ray diffraction method can be applied to small and medium-sized paper enterprises and other scientific research institutions with the development of science and technology.

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