Application Note

Measuring the surface potential and isoelectric point of polyester fibres with a zeta potential analyzer

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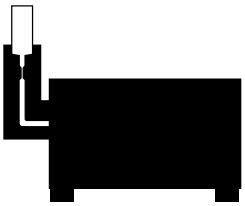
Polyester fibres are synthetic fibres manufactured from polyethylene terephthalate. Polyester fibres are widely used, not only in the textile industry. However, unmodified polyester fibres have deficiencies, e.g., a low water uptake, a hydrophobic character, and few reactive groups. This means they display a low affinity for dyes. Many techniques for improving the reactivity and hydrophilic character of polyester fibres exist in an industrial context. Generally, modifying fibres can enhance the interactions between the cations or anions of the dye and the functional groups on the fibre surface, and thus, the dyeability and water sorption of such fibres[1]. To track and analyse changes when developing new or enhancing existing fibre modification techniques, a sensitive measurement method is needed. A zeta potential measurement is a very powerful way to study the properties of surfaces[2]. It is able to evaluate the nature and dissociation of functional surface groups, and supplement contact angle measurements with additional information about the characteristics of the solid surface. Additionally, a zeta potential measurement is also able to evaluate the adsorption of ions and molecules. The ZPA 20 zeta potential analyzer from DataPhysics Instruments (Fig. 1) can measure the zeta potential of fibres, powders, and plate-shaped surfaces by means of a bidirectional streaming potential or streaming current technique. In this application note, we use the ZPA 20 to study the electrical properties and functional groups at the surface of polyester fibres.

Measurement device
Zeta potential analyzer



Measurement method
Oscillating streaming potential

Measured quantities
Zeta potential
Isoelectric point

Environmental conditions
Room temperature

Samples
Polyester fibre

Industries
Polyester fibre production
Synthetic fabrics production
Textiles industry
Textiles dyeing process

dataphysics
Understanding Interfaces

Technique and Method

Solid surfaces in contact with an aqueous solution are in most cases charged – by dissociation of functional groups and adsorption of ions and molecules from the solution. Even primarily uncharged surfaces in simple salt solutions usually carry a negative charge due to the adsorption of OH^- ions. If the solution moves with respect to the solid (or vice versa), a shear plane is formed between ions and molecules strongly adsorbed to the surface and the mobile ions in the surrounding solution (Fig. 2). The electrical potential at this shear plane, the so-called zeta potential, is a very sensitive measure for the charge situation on the solid surface. From pH- or concentration-dependent zeta potential measurements, conclusions can be drawn regarding the nature of the functional surface groups and adsorption processes[3].

The ZPA 20 zeta potential analyzer from DataPhysics Instruments uses the streaming potential (Eq. 1) or streaming current (Eq. 2) method to determine the zeta potential. An oscillating flow of an electrolyte solution is pumped either through a thin slit between two flat surfaces or the capillary system formed by a dense fibre or powder package. The solution flow shears off the mobile ion layer near the surface. As the ZPA 20 pumps the solution over or through the sample in both directions, its flow creates an alternating potential and current in the measuring cell. From the ratio of the streaming potential U_{str} or current I_{str} and the pressure difference Δp (Fig. 3), the zeta potential ζ is calculated[4][5]:

Fig. 1: The ZPA 20 zeta potential analyzer from DataPhysics Instruments

$$\zeta = \frac{\eta\kappa}{\epsilon_0\epsilon_r} \cdot \frac{\partial U_{str}}{\partial \Delta p} \qquad \text{(Eq. 1)}$$

$$\zeta = \frac{\eta}{\epsilon_0\epsilon_r} \cdot \frac{L}{HW} \cdot \frac{\partial I_{str}}{\partial \Delta p} \qquad \text{(Eq. 2)}$$

η is the viscosity of the solution, ε_r is the relative permittivity of the solution, ε_0 is the absolute permittivity of vacuum, κ is the electrical conductivity of the solution, L, H and W are the dimensions of the streaming channel between plate-shaped samples.

Data analysis using the streaming current requires knowledge of the dimensions of the streaming channel, which is usually not known for fiber or powder packings. These samples are investigated using the streaming potential method which allows calculating the zeta potential based on the viscosity and the conductivity of the solution.

Experiment

In this application note, the zeta potential ζ and the isoelectric point of a commercial polyester fibre sample were analysed using the ZPA 20 from DataPhysics Instruments (Fig. 1) with the streaming potential method.

To ensure the cleanliness of the measuring device and fibre measuring cell, the equipment was thoroughly cleaned with ultra-pure water (≤ 0.055 µS/cm) before preparing the measuring cell with the polyester fibre sample.

First, a set of a compression transfer disc and a compression disc were inserted into the fibre measuring cell MC-ZPA/PF and tightened lightly. A cluster of fibres was inserted evenly into the measuring cell using the pro-

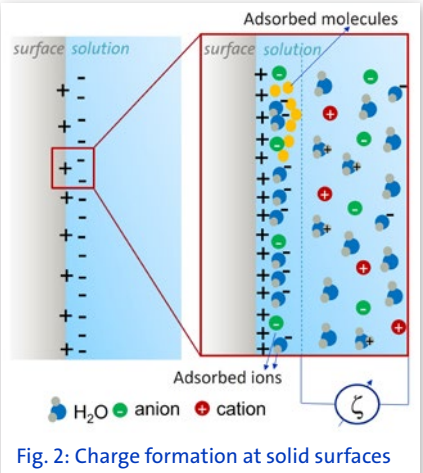


Fig. 2: Charge formation at solid surfaces in aqueous solution

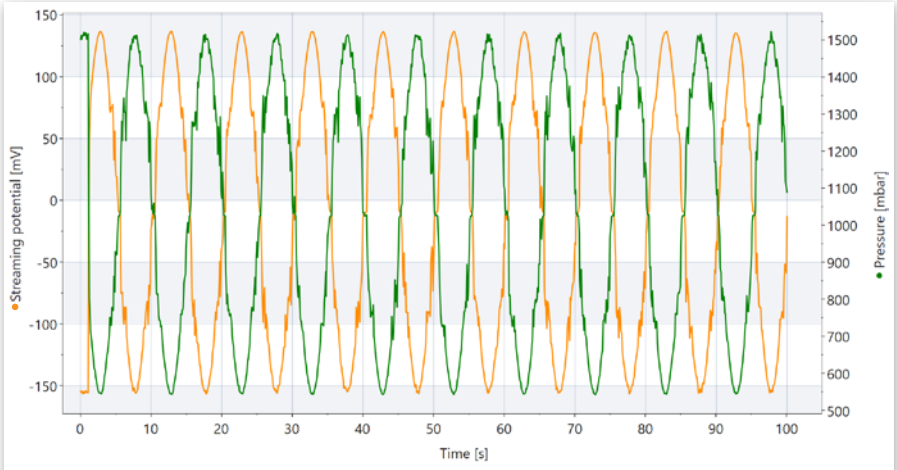


Fig. 3: Oscillating pressure (green) and resulting streaming potential (orange) vs. time

Fig. 4: MC-ZPA/PF measuring cell for fibres, powders, and granulate material together with sample preparation tools

vided stuffer (Fig. 4). Then, the second set of compression transfer disc and compression disc were inserted and tightened lightly.

After fixing the measuring cell to the ZPA 20, the storage vessel was filled with KCl solution (1 mmol/L, pH ~6). By using the 'bubble purging'-function of the ZPA 20, possible air bubbles on the sample and in the device's streaming channel were removed before starting the measurement. From the streaming potential vs. pressure ramps obtained in several oscillations (Fig. 3), the zeta potential was calculated for the given pH values. For each of the solution's pH value, a measurement time of only a few seconds is sufficient to generate results with excellent statistical quality. This underlines the usefulness of this new measurement approach, based on a bidirectional and oscillating solution flow.

Thanks to the automatic titration function of the LDU 25 liquid dosing unit from DataPhysics Instruments (Fig. 5), the zeta potential can be determined automatically in the pH range from 2.6 to 9.6. Titrations were done once from the neutral to the acidic range and once from the neutral to the alkaline range. As titrants, HCl (0.1 mol/L) and KOH (0.1 mol/L) solutions were used.

After the measurement, the device and the measuring cell were thoroughly cleaned, again using ultra-pure water. Cleaning is especially easy since no tubes are used in the ZPA 20 reducing the surface area and complexity of the parts tremendously.

Results and Discussion

Fig. 6 shows the zeta potential of the polyester fibre sample as a function of the pH value. The curve shows the typical decrease of the zeta potential from positive values at low pH values to negative values with increasing pH. In the case of the rather unpolar polyester fibers, this is partly an effect of the dissociation of functional groups, but mainly caused by the pH-dependent adsorption of H_3O^+ and OH^- ions. The measured values are consistent with published literature[2].

An important parameter for the verification of dissociable functional groups is the so-called isoelectric point, i. e. the pH value at which the zeta potential is zero. An isoelectric point below pH 4 and a plateau in the alkaline range indicate acidic surface groups. An isoelectric point above pH 5 and a plateau at low pH values are characteristic for alkaline groups. In Fig. 6, an isoelectric point at a pH value of 2.6 can be seen, showing the presence of acidic hydroxyl groups on the polyester fibre surface. The isoelectric point is consistent with the reported value for untreated standard polyester fibres[1], confirming the high reliability of the zeta potential measurement with the

Fig. 5: The LDU 25 liquid dosing unit allows operators to change the concentration inside the solution automatically.

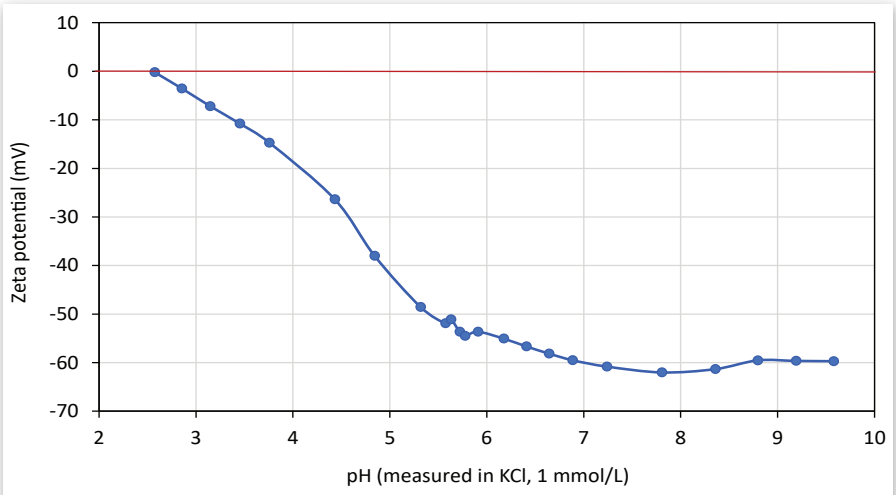


Fig. 6: Zeta potential of polyester fibre surfaces as function of the pH value

ZPA 20. Basically, polyester has only few dissociable groups. The acidic behavior of the studied fibres is rather an effect of sizings and processing aids applied during the spinning of the fibers. This proves that zeta potential measurements are highly surface-sensitive and indicate even small traces of surface coatings and contaminations.

A look at the measurement diagram in Fig. 3 shows that the bidirectional pressure ramps formed an even, sinusoidal curve, giving the operator a clear indication that the sample was prepared well and homogeneously. In view of the often-challenging preparation of a homogeneous sample layer for zeta potential measurements, the bidirectional approach easily reveals any issues that stay unnoticed when using unidirectional measurement techniques.

Summary

Using the ZPA 20 zeta potential analyzer from DataPhysics Instruments with its patented, bidirectional streaming potential or streaming current measurement method, the pH-dependent zeta potential and isoelectric point of polyester fibre surfaces were determined easily and reliably. Zeta potential measurements help to identify functional groups on the polyester fibre surface. This is very important in various application areas, e.g., the surface modification of polyester fibres to achieve higher dyeability, studying the adhesive behaviour between polyester fibres and coatings, or to prevent defects in the manufacturing process. This application note furthermore showcases the beneficial effects of the bidirectional measurement principle regarding sample preparation verification and measurement speed compared to other methods.

References

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