Experimental investigation on flow modes of electrospinning

Ting Si · Guang-Bin Li · Xing-Xing Chen · Rui-Jun Tian · Xie-Zhen Yin

Abstract Electrospinning experiments are performed by using a set of experimental apparatus, a stroboscopic system is adopted for capturing instantaneous images of the cone-jet configuration. The cone and the jet of aqueous solutions of polyethylene oxide (PEO) are formed from an orifice of a capillary tube under the electric field. The viscoelastic constitutive relationship of the PEO solution is measured and discussed. The phenomena owing to the jet instability are described, five flow modes and corresponding structures are obtained with variations of the fluid flow rate $Q$, the electric potential $U$ and the distance $h$ from the orifice of the capillary tube to the collector. The flow modes of the cone-jet configuration involves the steady bending mode, the rotating bending mode, the swinging rotating mode, the blurring bending mode and the branching mode. Regimes in the $Q–U$ plane of the flow modes are also obtained. These results may provide the fundamentals to predict the operating conditions expected in practical applications.

Keywords Electrospinning · Flow mode · Jet instability · Non-Newtonian fluid · Ultrafine fiber

1 Introduction

Electrospinning is an important method for producing ultrafine fibers. Different from traditional mechanical techniques, the electrospinning is referred to as an electrified cone-jet configuration [1–3], which is described as that under the force generated by a sufficiently large electric potential applied between an orifice of a capillary tube and a collector some distance away, a fluid of polymeric solution is electrified and a drop attached to the orifice approaches a cone. When the static electric force resulting from the charge on the surface of the cone surpasses its surface tension, an electrically charged jet is formed from the tip of the cone. At the same time, when the jet moves rapidly in a characteristic path and elongates under the electric field, the solvent evaporates. As a result, ultrafine fibers can be produced and novel fabrics and structures can be made according to various processes of nanofiber collections [1]. Because of the extremely small diameter and high ratio of surface area to mass, the electrospinning technique has gained extensive attention and has been applied in diverse fields including filter, drug delivery, clinical medicine, wound dressing, cosmetics, composites and others. The nanofibers produced by electrospinning are also advantageous in area of micromechanics, electricity and optics when particular materials such as pigments or carbon particles are added in the solutions. Besides, lots of topics such as the electrohydrodynamics, flow instability and non-Newtonian fluid are involved in the electrospinning process [3]. It is therefore of great significance to perform relative investigations.

To date a number of researches of the electrospinning have been done, containing hundreds of polymeric fluids and thousands of productions. The main progress can be found in Refs. [1–3] and references cited in, which provide access to comprehensive contributions. Nevertheless, most of previous researches pay much attention to the structure and morphology of single and cone-shell nanofibers and carbon nanotubes [4–7], but both the detailed process and mechanism of the formation of the cone and jet remain limited. For example, the phenomena such as branches on the electrostatically charged jet [8] and the bead-on-string structures [9–11] all cause damage in practice. Furthermore, the paths of the jet behave differently under different conditions. They are closely related to the physical properties and controllable parameters. Consequently, it is necessary to perform studies on the jet instability to meet the need in fields of scientific and engineering applications. Typically, in the electrospinning process the jet first moves with a straight segment.
and then bends into a three-dimensional coil and continues to elongate. Finally the thin jet solidifies into a nanofiber. The electric bending instability is considered as one of the main mechanisms [2, 3]. Taylor [12] first recognized and commented on the instability; Baumgarten [13] made some clear stop-motion photographs of the electrospinning jet. Subsequently, Reneker, Yarin and their co-workers [2, 3, 14–16] further studied the electrically driven bending instability taking the viscoelasticity of the polymeric solution, the evaporation of the solvent and the solidification of the fluid jet into consideration. The experimental results also indicate that the instability contains more than one type, i.e. there are the first, second and higher bending instabilities of the jet. Recently, theoretical and experimental investigations on the whipping instability of a micro-jet are further carried out and the results are helpful for understanding the mechanism of electrospinning [17, 18].

In the electrospinning process, the complex interplay of external electric field, free electric charge, viscoelasticity, surface tension and other factors may result in the competition of various flow modes of electrospinning. The downward growth rates and flow velocities of these modes are closely associated with the specific physical properties and controllable parameters. In this work, the phenomena owing to the jet instability and several flow modes and corresponding configurations of electrospinning are observed. For this purpose, a set of experimental apparatus of electrospinning is built up and a stroboscopic system is used to capture images of the cone and the jet. The aqueous solutions of polyethylene oxide (PEO) are utilized and their viscoelastic constitutive relationships are measured.

The paper is organized as follows. In Sect. 2, the experimental apparatus and materials are presented. The viscoelastic constitutive relationship of the aqueous solution of PEO is also given. The experimental process and characteristics of cone-jet configurations are briefly described in Sect. 3. The flow modes of electrospinning are classified in Sect. 4 and the behavior of the jet in each mode is described. Besides, in Sect. 5 some issues about this work are discussed. Finally, main conclusions are drawn in Sect. 6.

2 Experimental apparatus and materials

The experimental apparatus is sketched in Fig. 1. It involves two parts. One is the electrospinning generation device. The needle-plate structure is adopted as the electrode. The stainless steel capillary tube with the outer diameter of \( D_o = 0.7 \) mm and the inner diameter of \( D_i = 0.5 \) mm is linked to the anode of a high voltage DC power supply (ESSOP-20W/DDPM, 0 to 50 kV) and the round metal plate is linked to the ground as a collector. The confected PEO solution in water is injected precisely into the capillary tube by a syringe pump (WZS-5OF2, 0.1 to 99.9 mL/h). The other is the photograph capturing system. Due to small diameter and high velocity of the jet, a high speed video camera (FASCAM-SA5, 0 to 1 million fps) is used to capture instantaneous images of the jet. Specially, a simple but effective method is mainly utilized here, as sketched in Fig. 1. A stroboscopic lamp (PS-01A/B, 50 to 12 000 Hz) is used for illumination and a Fresnel lens (with the focus of 127 mm) is placed between the lamp and the jet. The images of the cone and the jet are captured by a microscope (SZ-B2/T2) combined with a CCD camera (MTV-1802CB) and a computer. The Fresnel lens working as a convex lens has wide surface and can concentrate the illumination on the position where the cone and the jet are formed. The center of the Fresnel lens is covered with a black paper to avoid high brightness of direct illumination of stroboscopic lamp and increase contrast of images. Note that the microscope has a limited resolution and a narrow focusing depth, while the jet is thin and has a three-dimensional envelope. A larger magnification of the microscope may result in a more amplified cone or jet but a more unsharp photograph. In general, the photograph capturing system should be appropriately adjusted in order to obtain clear and essential images.

\[
\gamma = \gamma_0 \sin(\omega t),
\]

Fig. 1 Sketch of the experimental apparatus

In the experiments, polymeric fluids such as solutions of PEO in water are utilized. Normally, their molecular weights \( M_r \) are in the range from several hundreds to several millions, concentrations \( W_r \) are from about 1% to 15%. Here we use a typical PEO solution with \( M_r = 500 000 \) and \( W_r = 5\% \), whose density is 1 034 kg/m\(^3\), conductivity is around 0.12 mS/cm. As the PEO solution is a viscoelastic fluid, whose flow characteristics are different from those of Newtonian fluids, it is of great significance to determine their viscoelastic constitutive relationships. Here the experiments are performed by using a Physica MCR301 rheometer (Anton Paar GmbH, Austria) that adopts plate rotor and small-amplitude dynamic measurement. The polymeric fluid is first filled in the space between the rotor and the fixed bottom plate, then the shear rate is forced on the fluid according to the rotation of the rotor. In this method a sinusoidal strain \( \gamma \) with an angular frequency \( \omega \) is applied, that is