Boron carbide (B₄C) nanofibers have a wide range of application in automobile and aircraft components, ballistic applications and nuclear power plants due to their high chemical inertness, thermal stability, hardness, excellent high temperature thermoelectric properties and neutron capture capacity. Since electrospinning technique enables mass production of one-by-one continuous nanofibers from various polymers, the electrospinning of B₄C nanofibrous membranes in the polymer provide large surface area to volume ratio, flexibility in surface functionality and superior mechanical and impact performance. In this study, by addition of polyvinyl pyrrolidone (PVP) into poly vinyl alcohol (PVA)/ boron carbide composite nanofibrous membranes, the effect of the electrospinning parameters on the fiber structure, morphology and its characteristic behaviour, depending on solution flow rate, applied voltage and distance between the substrate and syringe, were investigated by scanning electron microscopy (SEM) and FT-IR analysis.