The porous film considered in this study is the active layer of a novel nanostructured electrode for potential applications in electrochemical energy storage. For charge storage applications, it is important to disperse the active layer of the electrode on the current collecting substrate so that the electrolyte ions can diffuse easily, adding to the rate capability of the device. The dispersion of the active layer was accomplished during the fabrication of the electrode by first electrospray deposition of a carbonaceous precursor sol on Ni-foam, followed by curing the sol to form a rigid gel and subsequent carbonization. The electrospray process dissociated the sol into fine droplets at the time of deposition. The intent here is to develop carbon structure around the centre of the droplets, and accordingly the mesopores, which will enhance the diffusion of electrolyte ions in the active layers. Further, the carbon-coated nickel foam was subjected to electrodeposition in Nickel Nitrate Hexahydrate (Ni(NO3)2 .6H2O) solution, where Ni(OH)2 got deposited on the carbon surface. Finally, the coated substrate was calcined, leading to the formation of Nickel Oxide (NiO) layers over the carbon surface. The electrospray-induced nano-structuring of the active layer has been proved to be effective[23][24][25] in providing substantial rate capability alongside the density of energy storage. The diffusion co-efficient plays a pivotal role in the performance of these electrodes. There lies the motivation in working with the SEM images of this active layer, and studying the diffusion characteristics through estimation of effective diffusivity using a robust, accurate, and computationally inexpensive tool.