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Review:

A review of key parameters for effective electrophoretic deposition in the fabrication of solid oxide fuel cells*

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Abstract: This paper reviews recent progress in electrophoretic deposition (EPD), particularly in solid oxide fuel cells (SOFCs). EPD is a simple, cost-effective, and geometrical flexible colloidal process. With its excellent control of thickness and other morphological characteristics, it is favored for the fabrication of SOFCs because each component layer of an SOFC has different requirements. However, the effectiveness of EPD is closely related to the suspension stability and EPD processing parameters. Maintaining a stable suspension and optimizing the EPD processing parameters are essential to achieve a dense and uniform deposition layer. Key parameters in maintaining the suspension stability are generally categorized into colloidal related parameters, including particle size and solid loading, and suspension media related parameters, including dielectric constant and conductivity. The effects of these parameters are often reflected by the zeta potential of the suspension, which can be manipulated by using charging agents to maintain a stable state. The deposition time and applied voltage are key parameters in optimizing the EPD process through their effects on the deposition rate. The effects of these parameters on particle surface charges and on the EPD mechanism are discussed.

Key words: Solid oxide fuel cell (SOFC); Electrophoretic deposition (EPD); Suspension stability; Zeta potential; Colloidal
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1 Introduction

Solid oxide fuel cells (SOFCs) have gained increasing interest as an emerging power generation system that produces electricity through the electrochemical reaction between a fuel (e.g. hydrogen) and an oxidant (e.g. air) without combustion (Bariza et al., 2007; Li et al., 2009; Baharuddin et al., 2013; Mehmeti et al., 2016; Xiao et al., 2016; Masi et al., 2017). Fig. 1 illustrates the operation and components of an SOFC. Fuel is supplied to the anode and oxidized to release electrons, whereas oxidant is supplied to the cathode and reduced to oxide ions. These ions travel through the ion-conducting but electronically insulating electrolyte and react with hydrogen from the fuel to form water at the anode. The released electrons travel through an external circuit producing electricity. SOFCs have been used only for applications such as stationary power generation, hybrid power plants, trigeneration systems for high-rise buildings, and co-generation systems because of their high operating temperatures (>1000 °C)

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(Bieberle-Hütter et al., 2008; Fong and Lee, 2014; Buonomano et al., 2015; Tanaka et al., 2017). However, recent progress in thin-film technology and micro-fabrication has enabled the development of thin electrolytes that reduce the operating temperatures of SOFCs to intermediate (500–800 °C) and widen the choice of materials and the flexibility of fabrication methods (Mahmud et al., 2017; Singh et al., 2017), allowing the portable application of SOFCs (Evans et al., 2009; Mah et al., 2017).

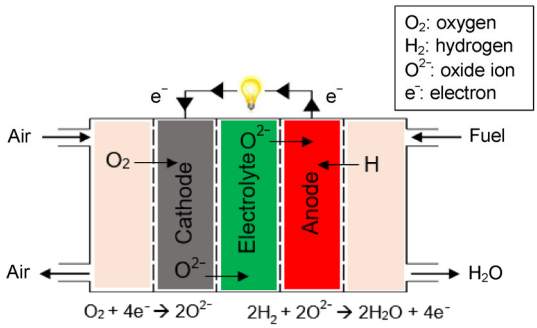

Fig. 1 Schematic illustration of SOFC operation and components

High fabrication cost is the main obstacle to the commercialization of SOFC systems (Piccardo et al., 2007; Irshad et al., 2016). The selection of a fabrication method should consider not only the cost but also the manufacturability and layer characteristics. Layer characteristics such as density and thermal expansion compatibility are closely related to the mechanical failure, power density output, and performance of SOFCs (Tietz et al., 2002; Minh, 2004; Blum et al., 2013). Among the established fabrication methods, such as screen-printing (Liu and Chen, 2009; Lee et al., 2010; Wu et al., 2012; Jalilvand and Faghihi-Sani, 2013; Somalu et al., 2017), tape-casting (Li et al., 2014; Schafbauer et al., 2014; Yu et al., 2015; Timurkutluk et al., 2016), plasma spray (Stöver et al., 2006; Han et al., 2016; Kumar et al., 2017), physical vapor deposition (PVD) (Collins et al., 2006; Marcano et al., 2017), magnetron sputtering (Rezugina et al., 2010), electrophoretic deposition (EPD) (Besra et al., 2007; de Riccardis et al., 2007; Santillán et al., 2009; Abdoli and Alizadeh, 2012; Irankhah et al., 2016), uniaxial pressing (Santillán et al., 2009; Kumar et al., 2012), dip coating (Lawlor, 2013; Menzler et al., 2013; Chen et al., 2015), and spin coating (Chen et al., 2012; Kim et al., 2016), EPD is of particular interest because of its versatility in fabricating complex geometry, short formation time, and easy control over layer thickness and morphology (Sarkar et al., 2004; Huang et al., 2008; Verde et al., 2012). Thus, EPD is deemed suitable for the production of tubular and planar SOFCs (Fig. 2).

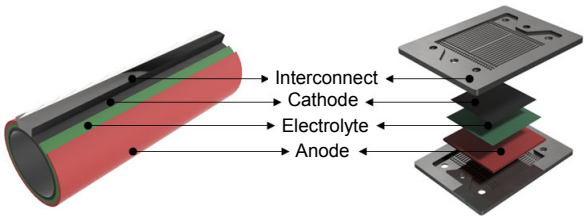

Fig. 2 SOFC tubular and planar designs

2 Electrophoretic deposition

An EPD system consists of a power supply, suspension, conductive substrate, and counter electrode (Fig. 3). The power supply creates an electric field that initiates the migration of the charged particles in suspension and their deposition on the conductive substrate (Corni et al., 2008; Diba et al., 2016). The electrode to which the particles move determines the type of EPD required and is represented by the sign of the zeta potential (Li and Zhitomirsky, 2008; Talebi et al., 2010; Chávez-Valdez et al., 2012; Ye et al., 2016). The positively charged particles form a layer on the negatively charged electrode in anodic EPD, and on the positively charged electrode in cathodic EPD (Fig. 4).

The mechanism of the EPD process has been explained by four theories: flocculation by particle accumulation (Hamaker and Verwey, 1940), particle charge neutralization (Grillon et al., 1992), electrochemical particle coagulation (Koelmans, 1995), and the electrical double layer (EDL) distortion and thinning mechanism (Sarkar and Nicholson, 1996). Among these theories, the EDL distortion and thinning mechanism is widely used today in explaining the EPD process. The mechanism of the EPD process involves three stages: double layer distortion, thinning of the double layer, and coagulation. The electrical double layer region of ions associated with a particle is distorted when the particle is forced towards a substrate material by an applied electrical

field. This distortion forms a 'tail' area which has a thicker double layer than the 'head' area of the particle (Fig. 5). As these counter ions in the 'tail' area react with the surrounding free ions when depositing on the substrate material, the double layer of the particle is thinned, allowing van der Waals (attraction) forces to dominate. The domination of van der Waals forces attracts surrounding charged particles which subsequently induce coagulation on the substrate (Besra and Liu, 2007).

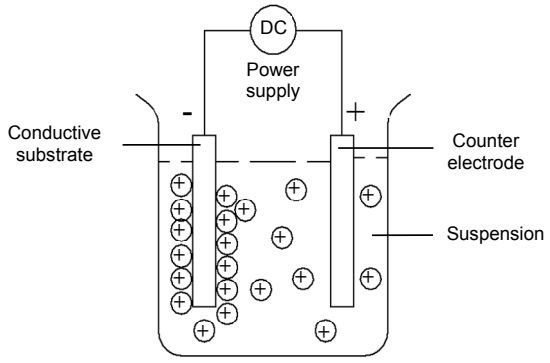

Fig. 3 Schematic illustration of the electrophoretic deposition system

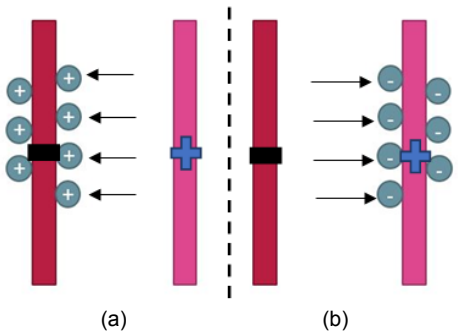

Fig. 4 Electrophoretic deposition type: (a) anodic; (b) cathodic

An electrolyte deposition layer on cathode material such as lanthanum strontium manganite (LSM) is not preferred due to the reaction between LSM and yttria stabilized zirconia (YSZ) electrolyte films, forming an insulating oxide layer at a high sintering temperature (Mahato et al., 2015). Given that the EPD technique was conventionally limited to conductive substrate materials, deposition of the electrolyte layer on an electronically insulating nickel oxide (NiO)-YSZ anode was not possible. Attempts to produce a conductive anode through a secondary process, like heat treatments in reduced atmospheres (Will et al., 2001) or the application of a conductive platinum coating (van Tassel and Randall, 2004) prior to the EPD process, increased fabrication costs. A breakthrough in depositing an electrolyte layer on the highly porous NiO-YSZ anode enabled the production of a low-cost electrolyte through the EPD process. Though non-conductive, the electrical charge carriers, usually electrons, in the suspension can easily flow through the pores with the application of voltage, thereby triggering particle movement and deposition (Besra et al., 2006). A highly porous NiO-YSZ anode can be fabricated through slip casting (Majhi et al., 2011), tape casting (Dayaghi et al., 2016), or uniaxial pressing (Baharuddin et al., 2016).

The EPD technique has proven successful in fabricating an SOFC comprising an anode, cathode, electrolyte, and a protective layer of the interconnect for tubular and planar designs. Cherng et al. (2013) fabricated a tubular SOFC through the EPD of all components on a thin copper wire. A YSZ electrolyte film was also successfully deposited onto a sufficiently porous non-conducting NiO-YSZ anode substrate using optimized suspension chemistry and

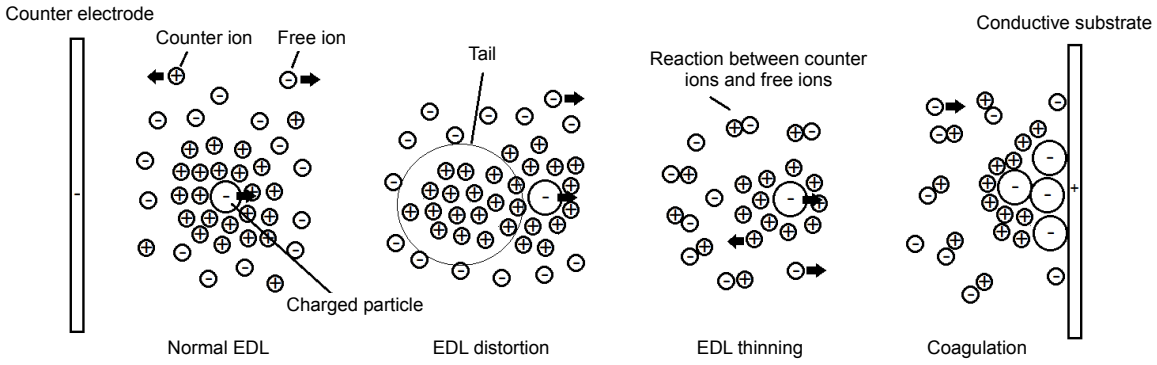

Fig. 5 Illustration of the EDL distortion and thinning mechanism

process parameters (Das and Basu, 2013). Moreover, the EPD technique can be used to deposit a thin copper manganese oxide (Cu, Mn, O) coating layer that ranges from 10 to 40 μm onto a Crofer 22 APU stainless steel interconnect plate to prevent the migration of poisonous chromium (Cr) gas to the cathode material (Smeacetto et al., 2015). The effectiveness of the EPD process is strongly influenced by suspension and deposition process parameters. This paper summarizes the key factors required to prepare a stable suspension, and the deposition process-related parameters involved in fabricating SOFCs through the EPD technique.

3 Suspension stability

A stable suspension, where particles have no tendency to agglomerate and have a slow settling rate, is important for the formation of a dense and strong adherence layer on the substrate (Besra and Liu, 2007). Agglomeration is influenced by attractive and repulsive interparticle forces. van der Waals forces attract the particles, whereas electrostatic forces caused by electrical double layers separate the particles. The suspension is stable and well dispersed when the repulsion between adjacent, similarly charged particles in the suspension exceeds the attractive forces (Bergström, 1997). Zeta potential measures the potential difference between a liquid medium and an interfacial double layer in the slipping plane, where high zeta potential indicates high repulsion between particles (Greenwood and Kendall, 1999) (Fig. 6). The influences of particle size, solid loading, suspension media, and charging agent on the magnitude of zeta potential are discussed in the following sections.

3.1 Colloids

Agglomerated particles may cause unstable EPD behavior (Hanaor et al., 2012) leading to high porosity and a sponge-like deposited layer that results in poor adherence to the substrate material (Krüger et al., 2004). Compared with agglomerated particles, well-dispersed particles provide a large contact area with free ions in the suspension, which increases the electrical double layer forces that bounce particles away from each other. The tendency of agglomerations is influenced by particle size, where small particle size results in a lower settling rate and better electrophoretic mobility during the deposition process than large particle size (Kalinina et al., 2016a; Matsuda et al., 2016; Suarez et al., 2016; Das et al., 2017; Sun et al., 2017).

Homogenizing methods, such as ball milling (Rahman et al., 2013), centrifugation (Kalinina et al., 2016b), or ultrasonification (Zhang et al., 2011), can be used to produce a fine and homogeneous particle size. For example, increasing the ultrasonification and centrifugation times of $Ce_{0.8}Sm_{0.2}O_{1.9}$ (CSO), $Ce_{0.8}Gd_{0.2}O_{1.9}$ (CGO), and $Ce_{0.8}(Sm_{0.75}Sr_{0.2}Ba_{0.05})_{0.2}O_{2-\delta}$ (CSSBO) produces fewer aggregated particles, reduces the span of particle size distribution, and increases the fraction of individual particles (Kalinina et al., 2016c). A similar result is observed when copper manganese oxide (Cu, Mn, O) is homogenized using the ball milling method (Sun et al., 2017). Increasing the ball milling time results in a fine particle size and well-dispersed particles.

Solid loading is a key parameter in producing a dense deposited layer. Increasing solid loading increases the deposition weight and produces a denser layer (Zarabian et al., 2013). However, high solid loading results in a high suspension viscosity, which is unfavorable for the EPD process. A suspension with sufficiently high solid loading and relatively low viscosity is ideal for successful dense layer deposition (Liang et al., 2007). Moreover, high solid loading tends to compress the double layer forces and increases the van der Waals attraction (He et al., 2013). Given that an increased attractive force increases agglomeration tendency, the repulsion between particles should be sufficiently high to maintain suspension stability and ensure a successful EPD even using high loading suspension. A suitable charging agent can be used to increase the repulsion between particles, that is, the zeta potential value. The application and effects of a charging agent are discussed later.

3.2 Suspension media

The suspension media can be either aqueous or non-aqueous; aqueous media are preferred in SOFC fabrication because their dielectric constant falls in the critical range between 12 and 25. A dielectric constant below 12 has a low dissociative power, whereas one over 25 reduces electrophoretic mobility

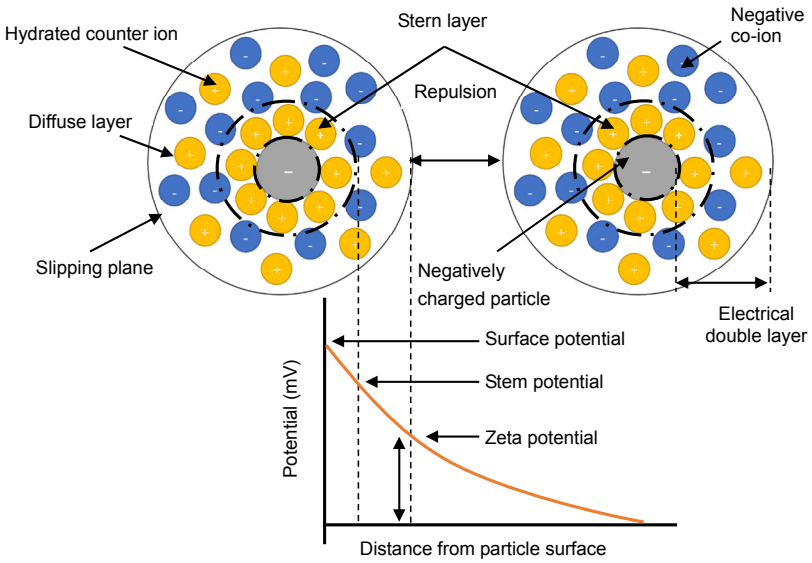

Fig. 6 Schematic of the double layer region of a particle

and the double layer region of the particles due to high ionic concentration (Powers, 1975). The different dielectric constants in each type of suspension medium create different interparticle forces and zeta potentials.

The optimum working range of conductivity for an effective EPD process is closely related to the properties of the suspension, particularly the zeta potential (Ferrari and Moreno, 1996). An excessively low conductivity leads to electronically charged particles that cause flocculation. An excessively high conductivity indicates a large number of free ions in the suspension, which become the main current carrier. The conductivity of a suspension increases as the ionic concentration, which indicates the number of free ions in the respective suspension, increases (López-Robledo et al., 2013). A high ionic concentration immobilizes the particles, reflecting a low zeta potential that is not preferred for an effective EPD process.

Zeta potential is the potential difference between the liquid medium and the interfacial double layer at the slipping plane, which is also known as the double layer region. Fig. 7 shows the zeta potential of various isopropanol-based suspensions in response to pH conditions based on previous research. Suspensions with relatively high zeta potentials are electrically stable, where the repulsion between adjacent and similarly charged particles in the solution exceeds the attractive forces. A magnitude of zeta potential above 30 mV is preferred to maintain a well-dispersed suspension. However, an excessively high zeta potential reflects an over-stabilized suspension caused by an excessively high repulsive force between particles. This hinders the formation of a layer on the electrode substrate during the EPD process, given that the electric field cannot overcome the repulsive force (Sarkar and Nicholson, 1996). High interparticle attractive forces in high solid loading suspensions result in a low zeta potential that is not favored for an effective EPD process. The zeta potential can be increased by manipulating the pH condition of the suspensions to maintain suspension stability.

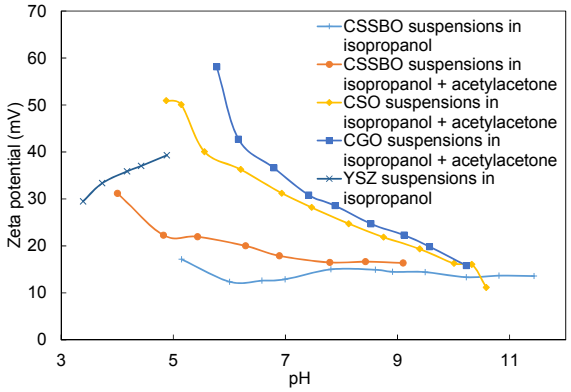

Fig. 7 Zeta potential of various isopropanol-based suspensions in response to pH value (Kalinina et al., 2016c; Das et al., 2017)

In general, the pH value is a measurement of the ionic concentration of H^+ and OH^- ions. A low pH (<7) indicates a high concentration of H^+ compared with OH^- ions. The pH condition of a suspension is strongly related to the colloidal material and type of suspension medium used. Table 1 summarizes the properties of various SOFC materials suspended in non-aqueous suspension media used in recent studies. All suspensions generally achieved a stable state at a pH value ranging from 4 to 5.8, which can be manipulated using a suitable charging agent. Even so, a suspension such as YSZ suspension in isopropanol combined with acetylacetone may not need a charging agent due to its high zeta potential. Such a suspension is called a self-stabilized suspension. A mixed suspension medium is preferable to a single component medium, such as YSZ suspension in isopropanol, which has a lower zeta potential (Kalinina et al., 2016b).

3.3 Charging agents

Charging agents can be used to manipulate the

Table 1 SOFC materials used in EPD with their respective component and suspension properties

Component	Material	Substrate	Suspension media	Charging agent	pH	Reference
Electrolyte	YSZ	NiO-YSZ	Isopropanol	Phosphate ester	4.8	Das et al., 2017
	CSO	$La_2NiO_{4+\delta}$ (LNO)	Isopropanol, acetylacetone	Potassium hydroxide	4.9	Kalinina et al., 2016c
	CGO				5.8	
	CSSBO				4.0	
	CSSBO	LNO	Isopropanol	Potassium hydroxide	5.0	Kalinina et al., 2016a
	YSZ	LSM	Isopropanol, acetylacetone	–	3.3–4.8	Kalinina et al., 2016b
	YSZ	NiO-YSZ	Acetylacetone	Ammonium salt of polyacrylic acid	–	Majhi et al., 2011
	LSO	Stainless steel	Ethanol	Polyethylene-imine	–	Suarez et al., 2016
Cathode	LNO	Gd_2O_3-doped CeO_2 electrolyte precoated with conducting polypyrrole	Ethanol	Butoxyethyl acid phosphate, phosphate ester, and polyethylene-imine	4	Matsuda et al., 2016
	LSCF	SUS304	Deionized water	Starch and polymethyl methacrylate	3	Rahman et al., 2011
	LSM/LSM-YSZ	YSZ	Acetylacetone	Iodine	–	Itagaki et al., 2012
	LSCF-SDCC	SDCC	Ethanol and deionized water	Poly diallyl dimethyl ammonium chloride	5	Baharuddin et al., 2016
Anode	Ni-YSZ	YSZ (painted with a graphite rod to yield a conductive surface)	Acetylacetone	Iodine	–	Itagaki et al., 2015
	NiO-YSZ	Stainless steel sheet	Isopropanol	Iodine	–	Zarabian et al., 2013
Interconnect coating	$Cu_{1.3}Mn_{1.7}O_4$	Crofer 22 APU	Acetone, ethanol	Iodine	–	Sun et al., 2017
	$Mn_{1.5}Co_{1.5}O_4$	Crofer 22 APU	Ethanol and deionized water	–	–	Smeacetto et al., 2015
	MnCoO	AISISAE430	Ethanol	Iodine	–	Mirzaei et al., 2016

pH condition to obtain a suitable zeta potential range for an effective EPD process. Charging agents are commonly available in bases, acids, or dispersants. The type of charging agent to be used is selected based on the initial pH value of the respective suspension. Each suspension offers a different trend of zeta potential in response to pH conditions. Compared with a non-aqueous suspension medium, aqueous suspension media, such as water, have a low isoelectric point (IEP), wherein the zeta potential value is zero (Fig. 8) (López-Robledo et al., 2013). Zeta potential shows no tendency toward IEP in a non-aqueous suspension medium because it has a smaller protolytic dissociation constant than an aqueous suspension medium (Kalinina et al., 2016a). The slower rate of reduction in zeta potential towards the IEP results in a lower tendency for agglomeration and polarity flip of particles in a non-aqueous suspension medium.

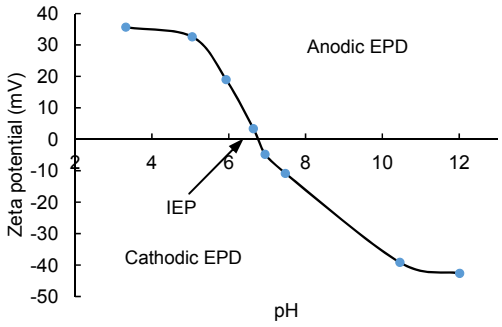

Fig. 8 Isoelectric point of gadolinium-doped ceria suspensions in water. Reprinted from (López-Robledo et al., 2013), Copyright 2013, with permission from Elsevier

The amount of charging agent used is important to ensure that all particle surfaces are charged to avoid agglomeration (Zarbov et al., 2004) and to control the polarity of the particles. Stringent control on the pH value is necessary to avoid reverse substrate deposition caused by changes in polarity during the EPD process.

In some cases, charging agents are also used to manipulate the suspension conductivity. Das et al. (2017) used phosphate ester (PE) to alter the conductivity of YSZ in an isopropanol suspension. The results showed that increased PE concentration increased suspension conductivity due to the rise in the number of H^+ ions. The H^+ ions could be absorbed into the double layer region causing an increase in the zeta potential value. Zeta potential stopped increasing after the double layer was covered completely by the ions (Fori et al., 2014). Increasing PE concentration increased the number of free ions, which is expected to intensify suspension conductivity and ionic concentration and consequently decrease the double layer region in the suspension (Kovalev et al., 2014). This characteristic leads to particle agglomeration or sedimentation due to a low zeta potential. High suspension conductivity can help increase particle mobility, but the zeta potential value must be prioritized to keep the particles well dispersed.

4 EPD process optimization

Both anode and cathode materials in SOFCs require optimum porosity to diffuse gas effectively (Itagaki et al., 2015; Gondolini et al., 2017), whereas the electrolytes must be sufficiently dense to separate the fuel in the anode from the air in the cathode (Yamamoto, 2000; Yang et al., 2012). Therefore, an understanding of particle mobility in the EPD process is essential to control the uniformity and density of the deposited layer to fulfill the morphological requirements of the components. The thickness and density of the deposited layer are influenced by the mass deposited during the EPD process. The higher the mass, the higher the thickness and density of the deposited layer (Das and Basu, 2014). Two key parameters need to be controlled during the EPD process, namely the applied voltage and deposition time. These parameters are strongly dependent on the zeta potential of the suspension, which is directly associated with particle mobility (Ferrari and Moreno, 2010).

Deposition time to achieve the desired deposited mass can be shortened by an increased voltage because higher voltage produces a stronger electrical field and subsequently increases suspension conductivity (Anné et al., 2004; Li and Zhitomirsky, 2009; Sakka and Uchikoshi, 2010; Fori et al., 2014). Thus, particle mobility increases and a layer on the substrate electrode forms faster with an improved suspension conductivity. Suspensions with low conductivity and high zeta potential can produce an effective EPD without altering the zeta potential value through the

supply of a suitable voltage (Ferrari and Moreno, 1997). However, an excessively low applied voltage causes slow particle mobility due to insufficient electrophoretic velocity to overcome the Brownian velocity of all particle sizes. Conversely, an excessively high applied voltage results in rapid particle mobility because of excessive electrophoretic velocity over Brownian velocity (Ovtar et al., 2012). The resultant deposited layers in both cases can be non-uniform (Smeacetto et al., 2015) and are not favored for the SOFC application.

Fig. 9 illustrates the electrophoretic deposition rate of nano-bismuth oxide (Bi_2O_3) suspended in isopropyl at various applied voltages. The deposited mass increases linearly as the deposition time increases in the early stage of the EPD process. After some time, the process slows down and eventually becomes saturated due to the formation of an insulating layer of ceramic particles on the deposition electrode. Further deposition will increase the discharge of ions from the deposited particles and raise the suspension conductivity. Thus, particle mobility increases, which promotes collision in different applied field strengths. Such collisions lead to sedimentation formation and flocculation which may cause gradient deposition and an unstable suspension (Guo et al., 2015).

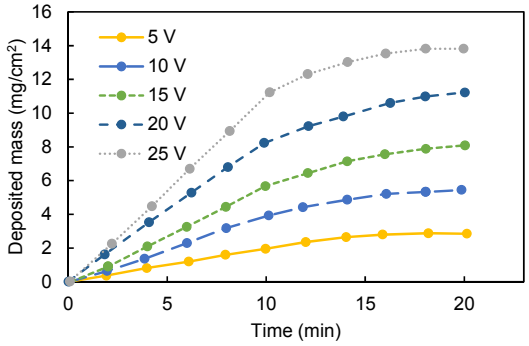

Fig. 9 Electrophoretic deposition rate of nano-bismuth oxide (Bi_2O_3) suspended in isopropyl alcohol at various applied voltages (Guo et al., 2015)

5 Conclusions

Despite the need to fabricate each SOFC component layer with desired properties and performance, the EPD technique facilitates the production of various SOFCs designs with low fabrication cost. However, suspension stability is a key parameter for achieving successful deposition. Recent studies on key parameters to maintain suspension stability for SOFC applications, including the particle size and solid loading of the targeted coating material and the dielectric constant and conductivity of the suspension media, are highlighted in this paper. Particle mobility is determined by the combined effect of the above-mentioned parameters, which can be quantified through zeta potential measurements. In short, a sufficiently high zeta potential is essential to ensure sufficiently mobilized particles for an effective EPD process. The zeta potential of a suspension can be tailored by manipulating its pH condition. Given that the layers of SOFC components involve various types of materials each requiring specific parameters to achieve effective deposition, careful preparation and optimization of the key parameters are essential to obtain the right recipe for each type of SOFC material.

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中文概要

题　目：电泳沉积固体氧化物燃料电池关键参数研究综述

目　的：电泳沉积是一种简单且具有成本效益的涂层技术。其出色的形态特征控制，适用于制造需要每个组件层都具有其独特属性的固体氧化物燃料电池。本文旨在综述电泳沉积的最新进展、制备稳定悬浮液所需的关键因素以及通过电泳沉积技术制造固体氧化物燃料电池所涉及的相关参数。

创新点：1. 分析了维持悬浮液稳定性的关键参数，包括粒径和固体载荷等胶体相关参数以及介电常数和电导率等悬浮介质相关参数。2. 讨论了这些参数对粒子流动性、电动电位和电泳沉积技术于固体氧化物燃料电池应用的综合效应。

方　法：1. 对以往的研究进行综述，并总结电泳沉积技术制造固体氧化物燃料电池组件层的发展（表 1），包括稳定悬浮液的制备以及电泳沉积工艺关键参数的优化。

结　论：鉴于每个固体氧化物燃料电池组件层都涉及不同类型的材料，且每种材料都需要特定的参数来实现有效沉积，因此，为了获得各组件层所需要的性能，制备悬浮液配方的正确性和电泳沉积工艺的优化显得至关重要。

关键词：固体氧化物燃料电池；电泳沉积；悬浮稳定性；电动电位；胶体