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KATA PENGANTAR

Assalamu 'alaikum warahmatullah wabarakatuh

Persaingan bisnis saat ini menunjukkan persaingan yang cukup ketat bahkan sampai pada persaingan global. Oleh karena itu industri dituntut untuk melakukan inovasi di semua aspek agar mampu mempertahankan tingkat persaingan. Jiwa kewirausahaan harus dimiliki oleh para pemangku kepentingan yang ada di industri untuk menghadapi ketatnya persaingan bisnis saat ini. Melakukan berbagai inovasi bisnis merupakan suatu keharusan bagi para wirausahawan (*entrepreneurs*), agar dapat mengkonversi tantangan/hambatan menjadi suatu peluang (*opportunity*).

Iklim bisnis di Indonesia dewasa ini ditandai dengan ketidakpastian dan penurunan kinerja sejumlah aktifitas bisnis. Hal ini disebabkan karena para wirausahawan belum memanfaatkan potensi yang ada secara optimal yang dapat digunakan sebagai modal dalam menghadapi persaingan, baik lokal maupun internasional. Indonesia memiliki potensi yang berdaya saing tinggi, sebuah peluang bagi para wirausahawan untuk mengolah potensi yang ada menjadi keunggulan bisnis.

Peranan pendidikan tinggi dalam membangun jiwa kewirausahaan menjadi cukup penting untuk dikembangkan. Sehingga pendidikan tinggi dapat dijadikan sebagai pusat pengembangan inovasi kewirausahaan maupun dapat dijadikan sebagai pencetak para wirausahaan yang unggul. Seminar Nasional ini dirancang untuk memberikan wawasan mengenai pentingnya mengembangkan peran *entrepreneurs* yang ada dan bila memungkinkan ditularkan kepada yg lain berkaitan dengan keberhasilan dan rencana pengembangannya. Beragam konsep, hasil pemikiran, dan hasil riset tentang kewirausahaan akan disajikan dan dibahas pula pada Seminar Nasional ini dalam mendorong tercapainya pembangunan berkelanjutan.

Sebagai sebuah forum ilmiah, seminar ini diharapkan sebagai media diseminasi informasi hasil penelitian dan perkembangan mutakhir antar pihak dengan berbagai latar belakang, mulai dari kalangan perguruan tinggi, lembaga penelitian, pemerintah/pengambil kebijakan, dan pihak industri. Diskusi antarpihak dengan berbagai perspektif ini diharapkan dapat memperluas *social networking* dan menghadirkan visualisasi yang lebih lengkap atas berbagai perkembangan penelitian di bidang teknologi industri, dan pada gilirannya diharapkan dapat memberikan kontribusi bagi perkembangan teknologi dan pemanfaatannya di Indonesia.

Atas nama Fakultas Teknologi Industri, Universitas Islam Indonesia, saya menyampaikan terima kasih yang sebesar-besarnya kepada semua pihak yang telah berkontribusi atas terselenggaranya Seminar Nasional Teknoin 2011 ini. Seminar ini dapat berlangsung karena usaha terbaik dari panitia pelaksana.

Wassalamu 'alaikum warahmatullah wabarakatuh

Yogyakarta, 19 November 2011
Dekan,

Ir. Gumbolo Hadi Susanto, M.Sc.

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Formulation of carbon ink for gas diffusion in fuel cell development

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Abstract

The Gas Diffusion layer fabrication for Membrane Electrode Assembly (MEA) Fuel cell used carbon ink. Carbon ink as parameter which is important in determine MEA Fuel cell is success in application. Carbon ink formulation was generally explained with conventional method but in this paper base on equation of correlation of polymer fraction with carbon particle size. Knowing model performance, used carbon ink which consists of PTFE, activated carbon, alcohol and water. The result of evaluation base on equation shows that formulation model can diagnose the active carbon needed about 5-20 micron and PTFE about 12.5 - 30 % W. for performance proton exchange membrane fuel cell (PEMFC)

Keywords: GDL, GDE, Current density, carbon ink, PEMFC

Introduction

MEA consists of the Gas Diffusion Layer and Gas Diffusion Electrode (GDE). Each of layers have different function so the materials was formulated. The materials are carbon ink, carbon cloth or carbon paper. In the article or journal, the carbon ink formulation no much knew detail a while design of carbon ink formulation as the main parameter for the success of MEA fuel cell. The carbon ink consists of Polymertetraflouroethylene (PTFE), Nafion, Carbon or Carbon-Pt, Alcohol and distillate water. Each of this materials influence properties of GDL or GDE fabrication. In GDL fabrication, Hsin at. al (2002) [1], altering the polymer concentration to generate the different of porosity so the GDL gives the different of current density. For carbon paper Hsin at. al used 30% PTFE for getting the high current. At the same time, [1,2], emulsify of carbon ink by using PTFE, Nafion and Vulcan. The Nafion concentration is 5% getting the best performance of fuel cell. Then, developed to fix performance looking at ionic conductivity by using PTFE materials. In 50% losses the high ohmic but the porosity can increase of 0.04 up to 1 [3,9]. So it is too with the others journal review needed PTFE 20 up to 50% [4,5,6]. In this paper, offered the simple of the formulation methodology to help calculation of the carbon ink composition in MEA fabrication for Fuel cell. Determine the chemical composition of carbon ink for the GDL with variable of polymer fraction, polymer thickness and particle size. The correlation will be get from calculation of the carbon ink composition for MEA Fuel Cell.

Theory

This present invention relates to a method of formulating an ink composition. In more particular, the present invention relates to a method of formulating an ink composition which will contain carbon particles and particulate polymer binders. Background of the Invention, A fuel cell is used for producing electrical energy from fossil fuels. The fuel cell will achieve higher efficiency as compared with an engine. Besides, the fuel cell is known to be environmental friendly with less pollution. One main component of the fuel cell is referred as a membrane electrode assembly. This component is important for producing electric in the fuel cell. Typically, an ink composition in the membrane electrode assembly can be formulated by a conventional method of trial and error. This method is difficult due to complexity of the probability theory.

There is some prior arts relating to various ink compositions and their formulation methods. A patent [9] discloses an ink constituent comprising at least one selected from dyes or pigments, at least one water-soluble solvent or water and at least one dispersant. The ink constituent that contains at least one A and at least one B of starting materials is represented by an equation of

$$D(AB) = (A_d - B_d)^2 + (A_p - B_p)^2 + C(A_h - B_h)^2,$$

where A_d and B_d are each a dispersion component of the surface energy, A_p and B_p are each a polar component and A_h and B_h are each a hydrogen bond component.

Another Patent [9] describes an aqueous dispersion of polymer particles containing a colorant, wherein the viscosity retention rate of the aqueous dispersion which is imparted by the colorant will be calculated according to a formula.

The formula shall be indicated as [viscosity retention] = [the 20-degree C 5 viscosity after preservation]/[20-degree C viscosity before preservation] x 100 that will give 75% or more but less than 115% of viscosity retention rate for an aqueous dispersion containing 4.4 mass percentage colorant and 10.0 mass percentage ethanol, 10 An ink jet ink containing a coloring agent and a solvent wherein the solvent will give a [Sigma]S which is defined by two formulas has been claimed in a Japanese [9]. The two formulas are indicated as

$$S = \text{HIM} \times 1000$$

And

$$[\text{Sigma}] S = S1 \times C1 + S2 \times C2 + S3 \times C3 + S_n \times C_n$$

where H is the total of hydroxyl group per solvent 1 molecule and the amino group, M is molecular weight of the solvent, SI 15 is the mass fraction in S of a solvent 1, Sn is the S of solvent n and Cn is the mass fraction in the ink of solvent n. It is desirable to invent a method of formulating an ink composition which will involve simple steps of calculation. Besides, it is important to discover a method of 20 formulating an ink composition which can provide high performance in a membrane electrode assembly. The ink composition shall be useful for applications in various industries such as painting, coating and decoration industry, catalytic industry and building industry.

The primary object of the present invention is to develop a method of formulating an ink composition which will involve simple steps of calculation. The presented formulae shall be easy to understand and apply for designing the ink composition. One of the objects of the present invention is to introduce a method of formulating an ink composition which can provide high performance in gas diffusion layer (GDL) and gas diffusion electrode (GDE) of the membrane electrode assembly. Moreover, the formulated ink composition shall be useful for applications in various industries such as painting, coating and decoration industry, catalytic industry and building industry. At least one of the preceding objects is met, in whole or in part, by the present invention, in which the embodiment of the present invention describes an ink composition containing carbon particles and particulate polymer binders wherein the weight properties of carbon particles and binders are determined by the following based on

$$x = \frac{\rho_c r}{3\rho_p t + \rho_c r} \dots\dots\dots(1)$$

$$y = 1 - \frac{\rho_c r}{3\rho_p t + \rho_c r} \dots\dots\dots(2)$$

- x = weight fraction of activated carbon ,
- y = weight fraction of Polymer,
- ρ_c = density of activated carbon and
- ρ_p = polymer density,
- t = polymer thickness and
- r = diameter of carbon particle

If y is the polymer composition PTFE and r diameter of particle of activated carbon so the composition correlation PTFE with binder thickness and diameter of activated carbon particle like in equation or figure 1 and 2.

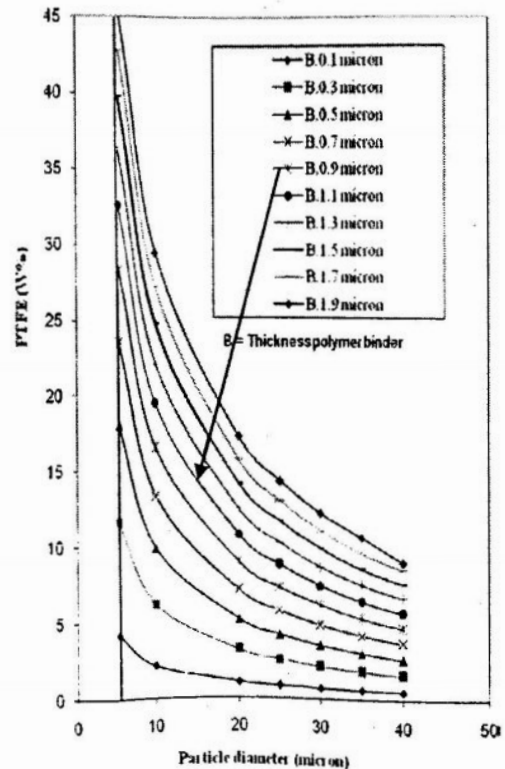


Fig 1. Diameter partikel carbon pada gas diffusion layer

Figure 1.. shown the connection diameter of carbon particles in the gas diffusion layer with PTFE as a binder percentage of carbon black particles. The larger of the particle diameter PTFE is required to bind the carbon particles are getting smaller. Presentation PTFE higher, then the thick bonds between PTFE particles higher. In some references mention the presentation of PTFE required 30 to

40%. In figure 2, tendency binder starts from 1 micron to 1.9 micron. Above 2.0 micron then the binder will be lower porosity of the coating. In figure 2, if the PTFE binder are considered ranging from 1 micron, then the tendency to use carbon black with a maximum particlesize of 15 microns.

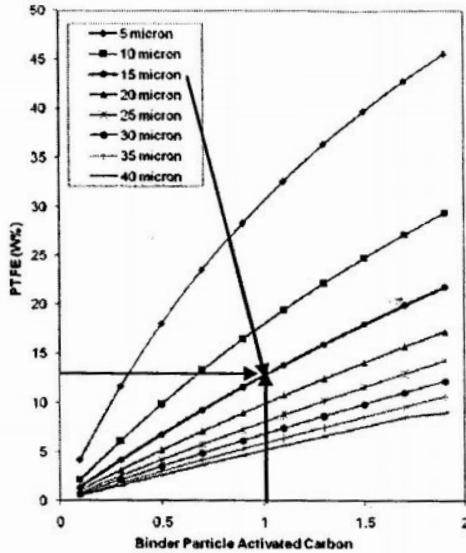


Fig 2. Binder Particle Activated Carbon

Therefore, for binding the particles of activated carbon is needed PTFE about 12.5% up to 30% weight and diameter of particle 5 up to 20 micron. For making the correlation polymer fraction and carbon toward the GDL, entering the water and alcohol into beaker glass and mixing in the turbulent area.

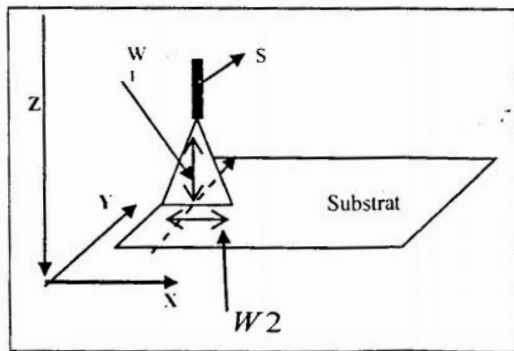


Fig 3. The x-y configuration

So enter activated carbon with particle size of about 15 microns (0015 mm) and weight percent of PTFE with 12.5% -30%. Each the result of mixing about 2 minutes is called the carbon ink GDL and Gde. Then the carbon ink is used to make the GDL through robotic spray method. The robot design

used in the system employs a specific attitude of expression of the x-y configuration [7,8] shown in Fig 3.

The spray variable is expressed by frequency (ω), nozzle height (W_1), distribution distance (W_2), division number of spray coating line on substrate (n) and nozzle velocity (S) [22]. The spray direction coating process is designed perpendicular to substrate. The nozzle frequency is given by equation 1.

$$\omega = \frac{nS}{2(ny + x)} \dots\dots\dots 1$$

In equation 1, the robotic frequency depends on S and N at certain boundary condition. If the nozzle height W_1 is direct proportional to Δx and W_1 is designed to be proportional with W_2 . Assumed the N_{spray} is known as the characteristic number of robotic spray, therefore the calculated value will be determined as follows based on the passing sprayer movement:

$$N_{spray} = \frac{S - 2\omega Sy}{2\omega W_1} \dots\dots\dots 2$$

Based on the equation 2, CAD is designed to possess the dimensions of x , y , n , S and W_1 level. The nozzle position on x-y axis is generated on the CAD system according to parameter n . The spray coating consists of the nozzle position and control code. The control code has a value corresponding to the desired substrate condition.

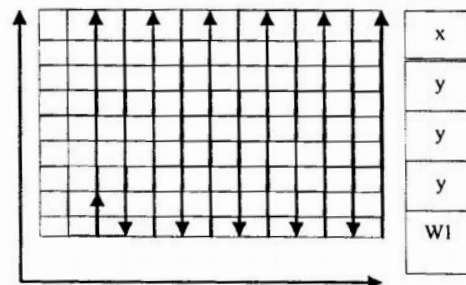


Fig 4. CAD of workpiece

CAD will produce a thick size (t_e), pore diameter (d_p), porosity (ϵ), typical activated specific surface (a_s). The variable of ink drop distribution of the nozzle will affect the layer size are μ , ν and p .

$$t_e, d_p, \epsilon, a_s = f(K, \mu, \nu, \sigma, p) \dots\dots\dots 3$$

Assume p is constant, surface tension (σ) constant, thus theoretically the correlation of t_e , d_p , ϵ , and a_s toward all variables and the robotic

movement as well as the drop variable are given by dimensionless equation 4

$$t_e, d_p, \varepsilon, a_s = f(N_{spray}, Re, W) \dots \dots \dots 4$$

The viscosity effect and surface tension are constant and neglecting the solidifying effect on substrate surface. Based on equation 3, the $t_e, d_p, \varepsilon, a_s$ are given by the dimensionless equation 4 to 5, as follows :

$$t_e, d_p, \varepsilon, a_s = f(N_{spray}) \dots \dots \dots 5$$

Equations 4 to 5, the t_e, d_p, ε and a_s of an electrode can be determined by the robotic characteristic number (N_{spray}).

Experiment Design

Gas Diffusion Layer (GDL) in MEA employed Vulkan XC-72R carbon black with particle aggregate 15 micron, PTFE 12.5-30 (w%), prophyl alcohol and carbon cloth with a thickness of 210 micron. Vulcan XC-72 loading in GDL is as large as 5 mg/cm². To obtain a sprayer characteristic, ink was spread over the GDL surface with N_{spray} values of 0.5 to 2.0. The spraying pressure of air mixture was 4 bar. The spraying applied hot plate under 60 °C to remove isopropyl alcohol. The flow chart of spraying is shown in Fig. 3 with operational condition as follows: nozzle height 7, hot plate under 60°C, air compressor 4 kg/cm². The pattern of nozzle 7 is shown in Fig. 4. The mixing process in mixing tank 5 is set to be laminair. The characteristic number of spraying is adjusted by PLC panel as desired. After spraying process, the substrate should be dried for 1 h and then put in the dryer under 80°C for 3 h. Afterwards hot pressure should be undertaken for 3 minutes, under 130°C and pressure of 50 kgcm⁻². This kind of work has been done for various numbers of spraying characteristic. The next step, examinations have been carried out for thickness, holes, hole diameter, surface area to obtain spraying data.

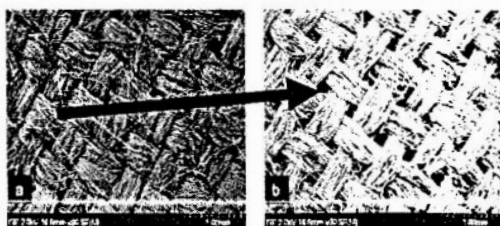


Fig 5. Carbon Cloth

- (a) Carbon cloth is soaked with PTFE and
- (b) (b) is a carbon cloth after immersion in PTFE used as a buffer

The instruments used for analysis to collect spraying data are the porosimeter and BET. Examination using instrument is employed for collecting data of holes, hole diameter and hole surface area. Fig. 5 adalah carbon cloth untuk penyanggah carbon black.

Result and discussion

The character of the influence of N_{spray} on the thickness and porosity as shown in equations and graphs to 22.5% of PTFE in carbon ink

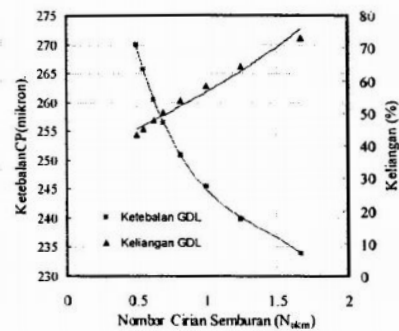


Fig 6. N_{spray} to the thicness and porocity of the GDL

$$\varepsilon = 0.37236 \exp[0.43 N_{spray}]$$

If the equation for the size t_e and deviation 12.4 %, the relationship was expressed by the equation.

$$\varepsilon = 0.37236 \exp\left[0.43 \left(\frac{t_e}{185.5}\right)^{0.1881}\right]$$

If the value of t_e is smaller, the ε value will be small This situation shows any t_e value , then the spray will give a certain ε value Formulations for 22.5% of PTFE by using mapping to the simulation results and various GDL research literature has a thickness (t_e) from 0:22 until 0:35 mm and ε from 55% to 70% [12,13,14,15,17,18,19]

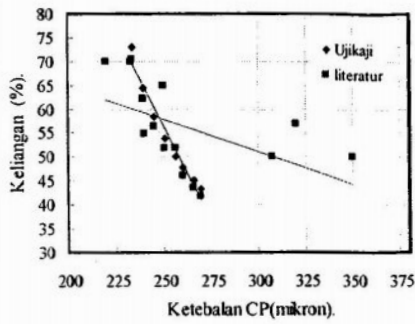


Fig 7. Porosity relationship with the thickness of the GDL

This graph shows the corresponding thickness of about 220 to 350 μ and porosity about 50% to 70%. This graph shows the porosity on the thickness of 220 to 270 μ . Achievement GDL is the same or better compared to GDL stated in the reference. Therefore, the optimization performance GDL through this formulation, carried out around the thickness of it. The relationship between the amount of PTFE with the character of the GDL as shown in the graph below. The instrument used to examine the pore volume, pore diameter and surface area using BET

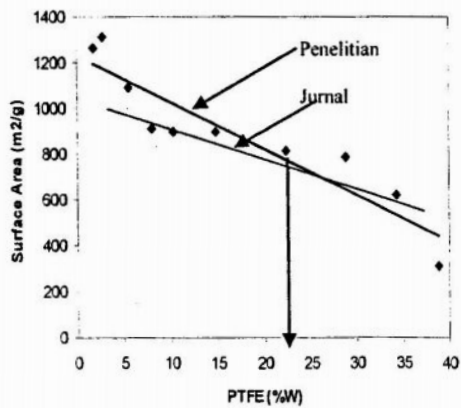


Fig 8. The characteristic correlation of the GDL with PTFE fraction

In the graph shown the results of a study published in the journal [12,13,14,15,17,18,19] and the results of this study formulations. Surface area is almost the same tendency. For the theory is built, binding the particles of activated carbon is needed PTFE is about 12.5% to 30% weight and particle diameter of 5 up to 20 microns. In the graph shows both the research and from various journals have a tendency surface area which is not much different. For PTFE 12.5% up to 35% weight, there is tendency formulation surface area is larger, meaning that it is

possible better use in fuel cell GDL. Figure GDL for 22.5 to 30 wt % PTFE which is very close to the mapping of the journal.

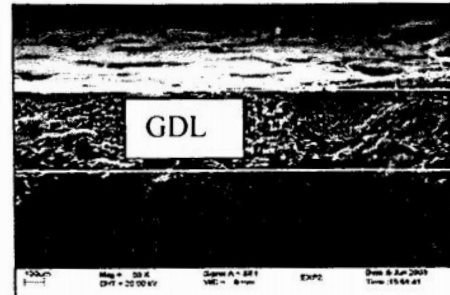


Fig 9. GDL dengan 22,5 % PTFE

Conclusion

The sprayer in this experiment can yield various sizes of thickness, diameter pore, porosity and activated specific surface area. Layer sizes can be performed with characteristic number robotic sprayer. Characteristic layer on substrate is close to characteristic simulation and experiment in journals.

From evaluation above can be gotten a conclusion that equation (1) and (2) can be calculated formulation of carbon ink. The correlation performance of polymer fraction with pore is same near with the performance pore with binder thickness. The formulation of carbon ink in the GDL fabrication need activated carbon about 5-20 micron (625-2500 mesh) and PTFE about 22-30 % weight.

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NOTASI

- N_{spray} = Number of robo sprayer
- p = pressure, bar
- R_e = Renould number
- S = speed nozzle, m/jam
- t_e = thickness, m
- $W1$ = nozzle height , m
- $W2$ = ink distribution width, m
- W_e = Weber number
- X, Y = substrate coordinate, m
- μ = viscosity, cp
- ϵ = porosity
- ω = frequency, 1/second

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Sertifikat

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Pemakalah

SEMINAR NASIONAL TEKNOIN 2011

MENGEMBANGKAN JIWA KEWIRAUSAHAAN YANG BERDAYA SAING
MENUJU PERSAINGAN GLOBAL


YOGYAKARTA, 19 NOVEMBER 2011

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