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## Enriching Engineering Science through Collaboration of Multidisciplinary Fields

**Bandung, Indonesia** • 28–29 October 2021

**Editors** • Istvan Farkas, István Seres, Józef Horabik, Zuzana Hlaváčová, Agus Saptoro, Husnul Azan Bin Tajarudin, Jono Suhartono, Mohammad Alexin Putra, Fahmi Arif, Arie Desrianty, Lauditta Irianti

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# Characterization of PES/CNTp-$TiO_2$ and PES/CNTp-ZnO composite membranes using solute transport method

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**Utari Puspa Kirana, Lisfia Nurhalisa, Mohammed Wali Hakami, et al.**

# Characterization of PES/CNTp-$TiO_2$ and PES/CNTp-ZnO Composite Membranes Using Solute Transport Method

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**Abstract.** Membrane technology can be applied in various industrial fields. Membrane characterization play significant role in membrane application to determine suitable membrane for the demanded process. In this study, the characteristics of (polyether sulfone) PES membranes with impregnated carbon nanotubes, $TiO_2$ and ZnO nanoparticles were investigated. The membranes were fabricated using phase inversion technique that involved *Dimethylformamide* (DMF) as solvent, DI water as coagulant and PES as the polymer. The resulting composite membranes were analyzed for its porosity, rejection, pore size and pore distribution where pore size and distribution was determined by solute transport method. The porosity analysis of pure PES membrane obtained was slightly higher than the PES/CNTp-$TiO_2$ and PES/CNTp-ZnO composite membranes, which are 42.9%, 35.55%, 33.4%, respectively. However, the resulting rejection of the solute diameter and molecular weight indicated that pure PES membranes had the lowest rejection values compared to PES/CNTp-$TiO_2$ and PES/CNTp-ZnO composite membranes. The membrane pore size distributions were among 0.1 to 50 nm where the highest probability of pore size for pure PES, PES/CNTp-$TiO_2$, and PES/CNTp-ZnO were 5 nm, 4 nm and 3 nm, respectively. These tests show that the addition of nanoparticles may increase the rejection value and reduce the pore size of the resulting membrane.

## INTRODUCTION

Membrane is a thin semipermeable layer that passes specific components and retains other elements of a fluid that is passed. Separation technology using membranes has the ability to move one component depending on the physical and chemical properties of the membrane and the resulting components. Displacement occurs due to the driving force in the feed in the form of pressure difference ($\Delta P$), concentration difference ($\Delta C$), electric potential difference ($\Delta E$), or temperature difference ($\Delta T$), as well as membrane selectivity which is expressed by rejection[1].

One type of membrane separation technology is ultrafiltration. Several polymers can be formed as ultrafiltration membranes, such as polyetersulfone (PES), polysulfones (PS or PSU), cellulose derivatives of acetic acid, polyacrylonitrile, or a combination of polymers. Polyethersulfone (PES) is the most commonly used polymer as a material for ultrafiltration membranes. PES is resistant to high temperatures, has wide pH operation range, has great mechanical strength, and is relatively easy in membrane fabrication[2].

The formation of membrane morphology such as membrane pores is strongly influenced by several factors, including using solvents, non-solvents, and types of polymers. The choice of polymer used can affect the performance of the membrane in the separation to be carried out because the resulting membrane structure affects the performance of the membrane. The problem that arises in the separation process using membranes is fouling, where fouling is an obstacle or impurity that can cause solute adsorption on the surface, which will clog the membrane pores known as pore blocking. This phenomenon must be considered in the manufacturing of membranes. Therefore, improving the performance of the membrane is very necessary to carry out the membrane modification process and method.

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The carbon nanoparticles addition may increase the average pore size of the membrane because the addition of hydrophilic in the casting solution causes the acceleration of solvent and non-solvent exchange, thus encouraging the formation of a more porous polymer structure[3]. on the other side, referring to Cao et al[4], the addition of $TiO_2$ into polyvinildene fluoride (PVDF) membrane have decreased the mean pore size of the resulting membrane. Therefore, this research studied membrane modification by incorporating nanoparticles into the membrane where impregnated titanium dioxide ($TiO_2$), and zinc oxide (ZnO) on carbon nanotubes (CNTp) were used to modify PES membrane. The membrane characterization was carried out, including the porosity of the membrane volume, the rejection coefficient, and the distribution of the membrane pore size with solute transport. The pore size limit for ultrafiltration membranes is around 1-100 nm[5]. Determination of the pore size distribution of ultrafiltration membranes is one of the keys to predicting and interpreting the rejection of different types of molecules[6].

# METHODOLOGY

## Materials

The polyetersufone (PES) polymer as main material for membrane fabrication was supplied by BASF, Germany. The others supported material such as pristine carbon nanotubes (CNTp), N,N-dimethyl formamide (DMF) solvent, titanium dioxide ($TiO_2$), zinc oxide (ZnO) were bought from Sigma Aldrich, Singapore. Whilst for sodium hydroxide, hydrochloric acid, polyethylene glycol and polyethylene oxide were bought from Merck, Indonesia.

## Experimental Method

### *Nanomaterial Impregnation*

The impregnation of titanium dioxide and zinc oxide was done by referring to Ashkarran et al[7]. firstly by poured 75 mL of DMF solvent into a 100 mL beaker glass followed by addition of 0.02 grams of CNTp and 0.1 grams of $TiO_2$ nanoparticles into the glass. The glass containing DMF, CNTp, and $TiO_2$ then placed in a UV light box for UV illumination with slow stirring at room temperature for 2 hours. The similar process was done for zinc oxide impregnation.

### *Dispersion of Nanomaterials*

The impregnated solution obtained from step 2.2.1 was ultrasonicated on ultrasonic bath at a frequency of 38 kHz for 1 hour. This process was divided into 4 stage where every stage was conducted by 15 mins ultrasonication and 5 mins cooling to ensure the solvent temperature will not exceed 30'C.

### *Nanocomposite Membranes Preparation*

Prepare 4.12 grams of PES powder and poured into 50 mL beaker glass, a 30 mL of dispersed CNTp-$TiO_2$ solution was then poured into the glass. The mixed solution was stirred at 250 rpm for 3 hours and the temperature was controlled at 70°C. Once homogeneous solution obtained, the solution is put in a desiccator to ensure no air bubbles remain in the solution. The ready solution was then casted in a smooth and clean glass plate using a doctor's blade with a thickness of 200 μm. The casted solution was immersed in a coagulation bath for 2 hours to ensure the polymer solution has turned into a membrane completely.

### *Separation of PEG/PEO*

Standard solution of PEG/PEO with a concentration of 1000 ppm was prepared by dissolving 1 gram of PEG/PEO into 1000 mL of distilled water. The molecular weight of PEG/PEO used was 3.35 KDa, 10 KDa, 35 KDa, and 100 KDa. The solute separation was initialized by pouring the PEG/PEO feed solution into the feed tank as much as 500 mL. The feed was then pumped into the membrane to obtained the sample of permeate. The permeate is analyzed for its PEO/PEG concentration.

### Membrane Characterization

In the membrane characterization, several tests were carried out, namely:

*Membrane Volume Porosity*

Membrane volume porosity is defined as the ratio between the volume of pores and the total volume of the porous membrane. The volume of the pores is determined by measuring the volume of water that occupies the pores of the wetted membrane using the difference in mass between the wet and dry membranes. So it can be found by the following equation:

$$\varepsilon = \frac{m_l/\rho_l}{m_l/\rho_l + m_m/\rho_m} x\ 100\% \quad (1)$$

Where $m_l$ = mass of liquid, g; $m_m$ = mass of the membrane, g; and $\rho_l$ = density of water, g/mL; and $\rho_m$ = density of membrane, g/mL.

*Rejection Coefficient*

The membrane's selectivity to the mixture is generally expressed by one of two parameters, namely the rejection coefficient (R) and the separation factor (α). A mixture of a dilute solution consisting of a solvent (mostly water) and a solute is more suitable for the solute's retention. The solute is partially or completely retained while the water solvent molecules freely pass through the membrane. Rejection is expressed in the equation:

$$R = (1 - \frac{C_p}{C_f}) x\ 100\% \quad (2)$$

Where R = rejection coefficient, %; cp = permeate concentration, g/mL; and cf = feed concentration mg/L.

*Solute Transport*

The solute transport test continues the ultrafiltration test, where the feed is a solution of various solutes whose molecular weight is known. PEG and PEO polymers were selected for the solute transport assay because they are easy-to-read synthetic polyethers and are available in various molecular weights. What's more, PEG and PEO are water-soluble.

For PEG

$$a = 16{,}73 \times 10^{-10} M^{0{,}557} \quad (3)$$

For PEO

$$a = 10{,}44 \times 10^{-10} M^{0{,}587} \quad (4)$$

From the empirical equations (2.17) and (2.18), Stoke's molecular radius of PEG and PEO can be obtained based on their molecular weight. The membrane pore size distribution can be expressed by the probability density function, which the following equation can express.

$$\frac{df(dp)}{d\,(dp)} = \frac{1}{dp \ln \sigma_p \sqrt{2\pi}} \exp\left[-\frac{(\ln dp - \ln \mu p)^2}{2(\ln \sigma_p)^2}\right] \quad (5)$$

Where = pore size, = geometric standard deviation, and = average pore size. The mean pore size ($\mu p$) was obtained from 50% solute separation, while the geometric standard deviation ($\sigma p$) was obtained based on the ratio of 84.13% and 50% solute separation[8].

## RESULTS AND DISCUSSION

### Membrane Volume Porosity

In this study, membranes were made by phase inversion immersion precipitation. Three membranes were produced namely pure PES, PES/CNTp-$TiO_2$ membrane, and PES/CNTp-ZnO membrane. The porosity of membranes is shown at table 1.

**TABLE 1** Porosity of Membranes

| Membran Type | Porosity (%) |
|---|---|
| Pure PES | 42,93 |
| PES/CNTp-$TiO_2$ | 36,57 |
| PES/CNTp-ZnO | 33,40 |

**Table 1** shows the value of the results of the membrane porosity measurement. It can be seen that the porosity value of pure PES membranes tends to be greater than the modified membrane porosity values (PES/CNT-$TiO_2$, and PES/CNT-ZnO). However, among the membranes, the porosity of PES/CNT-ZnO membrane was the smallest compare to the other membranes. The resulting smaller porosity indicates that the empty space on the membrane is getting narrow. The additions of nanomaterials have lowered the PES membrane porosity by up to 15% and 22% for CNT-$TiO_2$ and CNT-ZnO, respectively. This indicated that the addition of nanomaterials affects the porosity of the membrane.

PES is a hydrophobic material, thus when contacting with the polar non-solvent material in the coagulation bath, the non-solvent tends to hinder the contact with this hydrophobic polymer. This phenomenon will lead to development of a bigger space or pore in the membrane. moreover, the membrane porosity might also be affected where more space in the pure PES membrane lead to bigger porosity. Meanwhile, the addition of CNTp and $TiO_2$/ZnO nanoparticles into PES makes the membrane more hydrophilic since ZnO, and $TiO_2$ nanoparticles can improve the hydrophobic properties of PES. If viewed based on the manufacture, the process of membranes fabrication with the addition of nanoparticles is carried out by an impregnation process using UV, this process may also assist the hydrophilic properties of the membrane. The polar–polar material will tend to attract each other, thus resulting in the less empty space formed or dense.

The PES/CNT-ZnO has lower porosity compared to PES/CNT-$TiO_2$, this is because of the hydrophilic properties of ZnO are better than $TiO_2$. Garner, et al[9] investigated the contact angle of ZnO and TiO using a different fluids, they found that contact angle of ZnO was around 63$^o$ whilst for $TiO_2$ (21nm diameter) was around 67$^o$ and $TiO_2$ (100nm diameter) was around 79$^o$ for water as drop fluid. As explained above, the hydrophilic compound interactions will lead to high attraction between each other, so that the proportion of space in the membrane will become denser. As a result, porosity of the PES/CNTp-ZnO membrane is smaller than that of the PES/CNTp-$TiO_2$ membrane. This can prove that the modification of PES membranes by CNTp (Carbon nanotubes) and nanoparticles ($TiO_2$ and ZnO) can reduce the empty space on the membrane so that the porosity value will be smaller when compared to pure PES membranes.

## Rejection Coefficient

Rejection coefficient can be interpreted as the ability of a membrane to be able to withstand a component. Determination of the rejection coefficient was measured by UV-VIS spectrophotometer against the test sample. The test sample used is the permeate result of PEG/PEO solution with different PEG/PEO molecular weight sizes used. Following are the results of the rejection coefficients on various types of membranes.

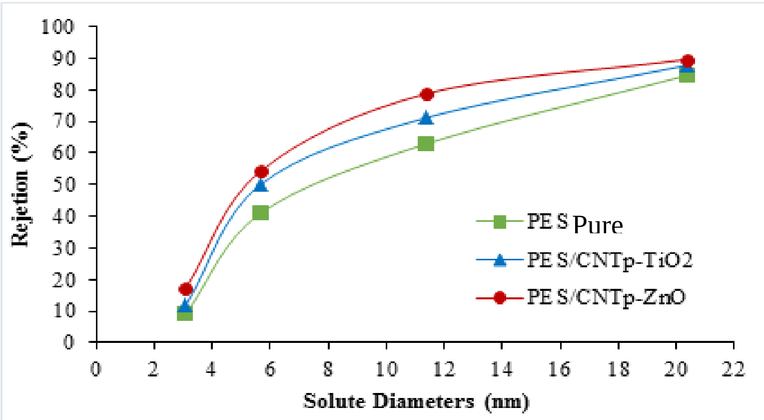


**FIGURE 1** Relation between Solute Separation and Solute Diameter

Figure 1 shows that bigger diameter of solute resulted in greater value of percentage separation of the solute. Bigger diameter of the solute will yield in more solute rejected by the membrane. In contrary, smaller diameter of the solute produced in more solute passing through the membrane. The value of percentage separation of the solutes can be known through the absorbance value in the sample solution that passes through the membrane where the PEG/PEO were used as solutes. Sample was carried out every 5 minutes. Figure 2 shows the relation between solute separation and solute molecular weight.

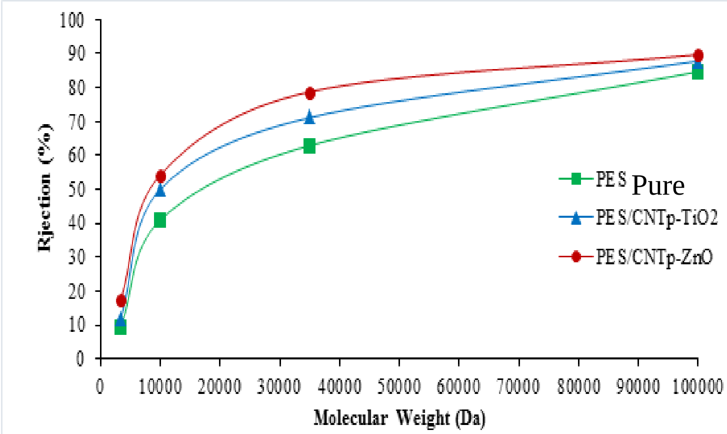


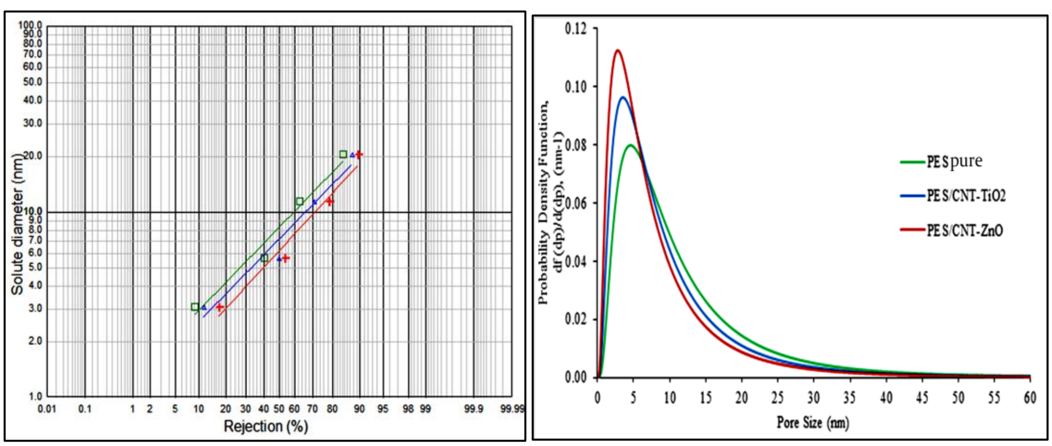

**FIGURE 2** Relation between Solute Separation and Molecular Weight

Figure 2 shows that bigger solute molecular weight yield in stronger rejection by the membrane. Thus, a significant solute rejection means that the membrane able to reject solutes or hold most of unwanted solutes to penetrate the pores. Pure PES membranes have the rejection of 84.65%, while membranes with modified nanoparticles have rejection values of 87.81% and 89.67% for PES/CNTp-$TiO_2$ and PES/CNTp-ZnO, respectively. The membrane without the addition of nanomaterials or pure PES membranes had the lowest rejection compared to membranes with the addition of nanomaterials, namely PES/CNTp-$TiO_2$ and PES/CNTp-ZnO membranes. The addition of nanomaterials able to improve the membrane structure to be denser thus increased the rejection.

Based on the addition of the type of nanoparticles, PES/CNTp-$TiO_2$ membrane has a smaller rejection compared to PES/CNTp-ZnO membrane. This is based on its nature, ZnO nanomaterials are more hydrophilic so that when the composite membrane solution is put into a coagulation bath containing aquadest, the ZnO nanoparticles will tend to attract to create a tighter pore structure. When the pore structure gets tighter, the rejection ability of the membrane gets bigger. ZnO nanomaterials have better hydrophilic properties when compared to $TiO_2$, so the rejection ability of PES/CNTp-ZnO membranes is greater than PES/CNTp-$TiO_2$ membranes.

## Solute Transport

Membrane characterization based on Solute transport data was produced by passing PEG and PEO solvents to the membrane using a pressure of 4 bar, then the permeate sample was obtained from the filtration results. Permeate samples from each membrane were then analyzed using a UV-Vis Spectrophotometer with a wavelength of 192 nm, and absorbance data were obtained from each sample. The rejection value was obtained from the absorbance data, then plotted into a logarithmic graph.




(a) (b)

**FIGURE 3** (a) Logarithmic graph of rejection of solute diameter; Green is Pure PES, Blue is PES/CnTp-$TiO_2$, Red is PES/CnTp-ZnO. (b) Relation between probability density and pore size.

From figure 3 the value of solute diameter at 50% and 84.13% rejection to determine the geometric standard deviation can be obtained. The probability of each membrane pore size can be calculated from equation 2.5. Based on calculations using these equations, a graph of the relationship between probability and membrane pore size is obtained, as shown in the figure 3b.

Figure 3b shows that the possibility distribution of pore sizes for pure PES membranes are around 0.3 nm to 58 nm with the highest probability of pore size occurred at 4.5 nm. Meanwhile for PES/CNT-$TiO_2$ membranes, the possibility size distributions are around 0.1 nm to 50 nm with the highest probability of pore size at 3.5 nm and for PES/CNT-ZnO membranes, the possible size distribution of PES/CNT-ZnO membranes are around 0.2 nm to 45 nm with the highest probability of pore size at 2.8 nm. Based on the average pore diameter, pure PES membranes have an average pore size of 5.3 nm, whilst for PES/CNT-$TiO_2$ and PES/CNT-ZnO membranes slightly smaller that are 4.3 nm, and 3.2 nm, respectively.

Based on these data, pure PES membranes have larger pore sizes than membranes made with nanomaterials. This proves that the addition of nanomaterials produced in smaller size of the membrane pore size. The hydrophilic properties may play an important role in pore size formation where hydrophilic compound are more likely to interact with polar compound. Similarly, PES/CNTp-ZnO and PES/CNTp$TiO_2$ composite membrane solutions when contacted with a polar aquadest will tend to be easily attracted. Meanwhile, pure PES membranes have larger pore sizes, this might be occurred due to PES has hydrophobic properties where the polimer hindered the attraction with the polar aquadets. In addition, the highest probability of pore size for PES/CNTp-ZnO are smaller compared to PES/CNTp$TiO_2$. This phenomenon might also supported by Garner, et al[9] where the hydrophilicity of ZnO were higher compared to $TiO_2$. Based on the average contact angle value obtained from the results of this study, it can be proven that ZnO has better hydrophilic properties than $TiO_2$. It has been acknowledge that when a surface has a contact angle less than 90°, the surface will tend to be hydrophilic. Meanwhile, when it has a contact angle greater than 90°, then the surface will have more hydrophobic properties. Therefore, smaller contact angle formed on the membrane surface, the better the hydrophilic nature of the membrane.

## CONCLUSION

This research have studied the characterization of PES membranes with nanomaterial TiO2 and ZnO impregnation in pristine carbon nanotube. The membrane characterization was conducted by solute transport method. The presence of $TiO_2$ and ZnO on the PES membranes affected the membrane porosity and rejection character. Based on the percentage of porosity obtained, it shows that pure PES membranes have a greater percentage of porosity when compared to the porosity of membranes impregnated with nanomaterials.

The presence of nanomaterial in the PES membrane has also increased the solute rejection from 84.65% to 89.67%. The increasing performance of impregnated membrane was believed as the result of the hydrophilic interaction between membrane and the polar coagulation non-solvent used during membrane formation. It is also found that the pore size of PES/CNTp-$TiO_2$ and PES/CNTp-ZnO membranes were smaller compared to pure PES membrane. The highest probability of membrane pore size for PES/CNTp-ZnO and PES/CNTp$TiO_2$ are 3.2 nm and 4.3 nm, respectively. Whilst for pure PES membrane is 5.3 nm.

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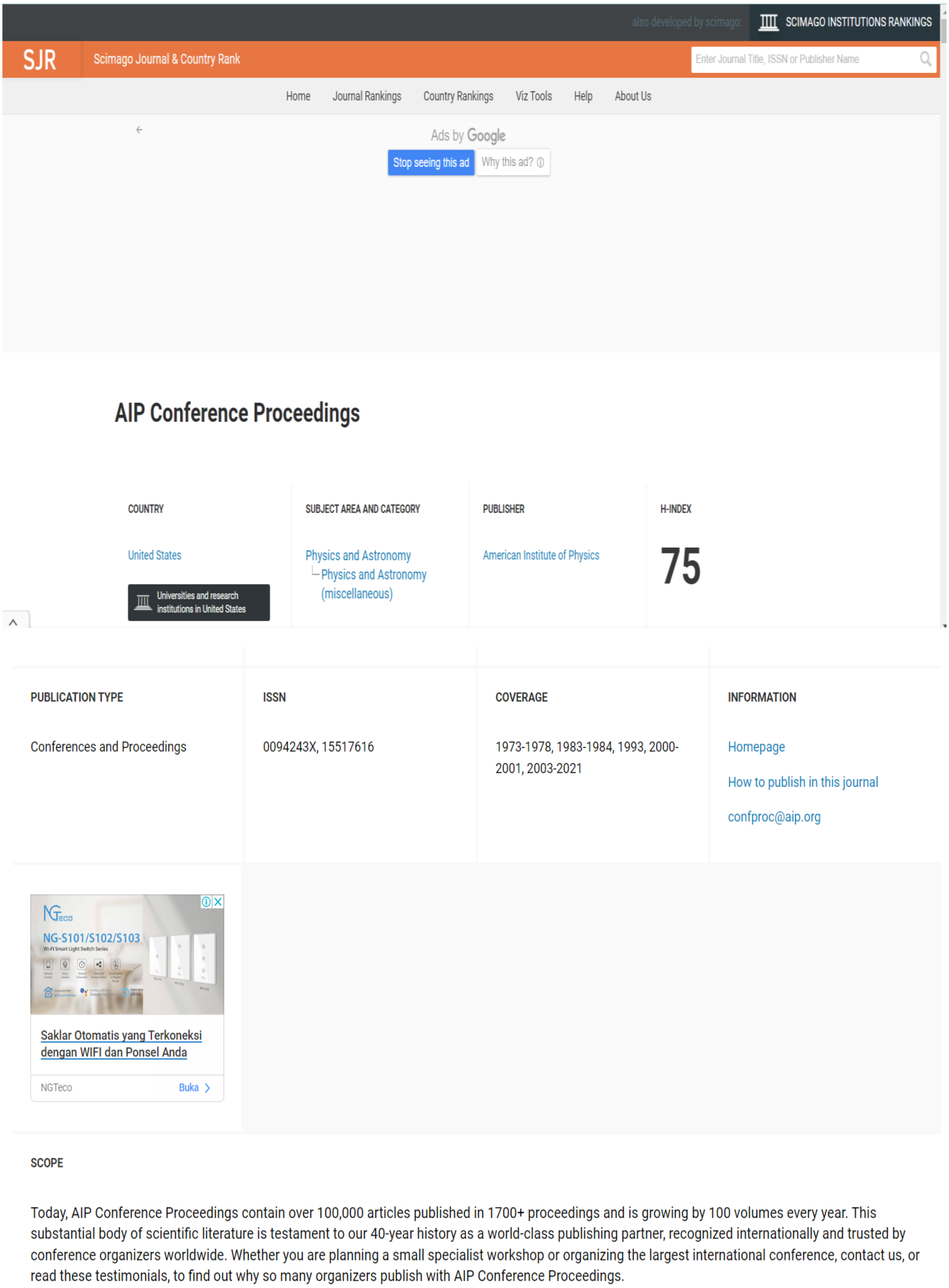

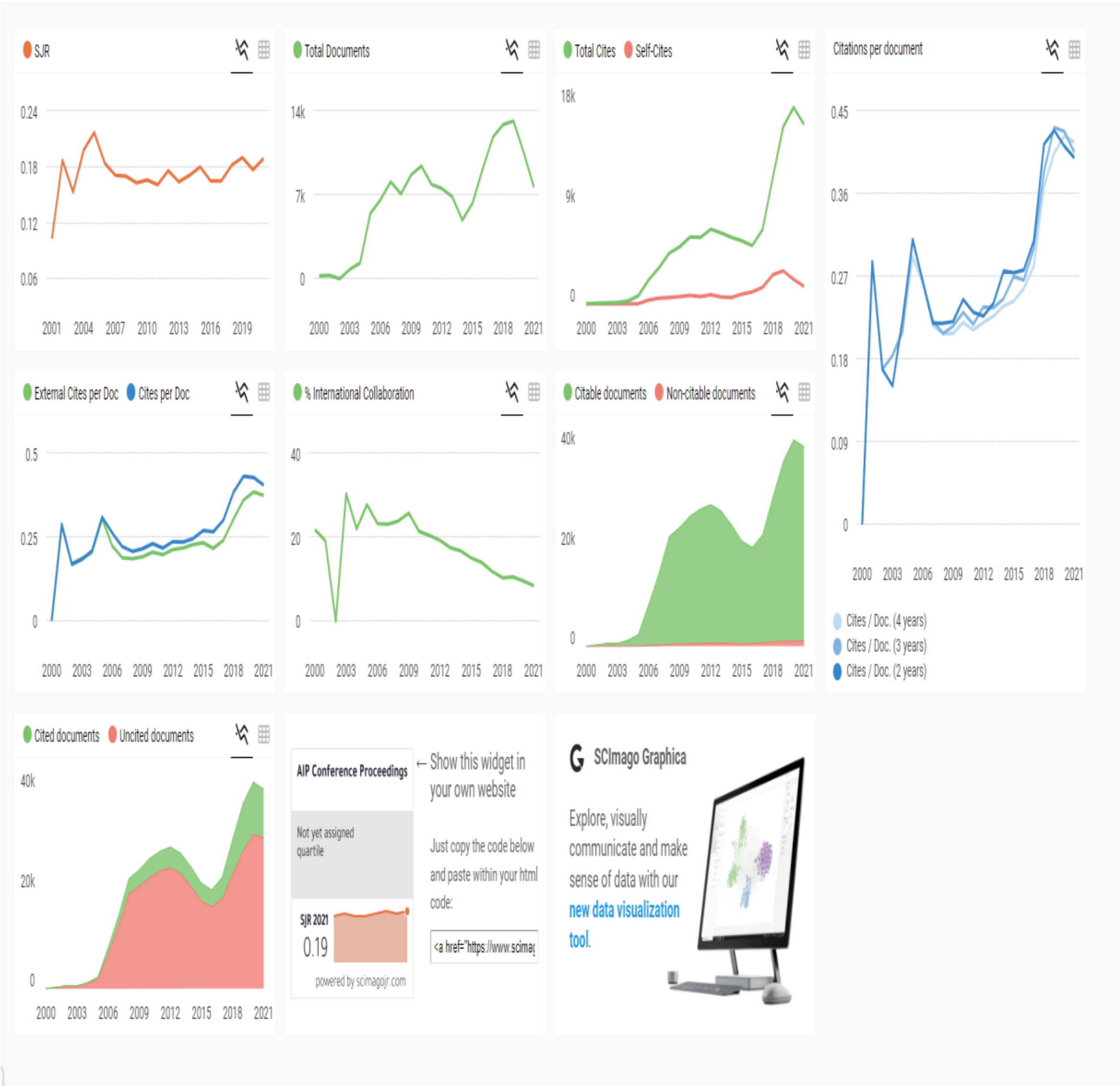
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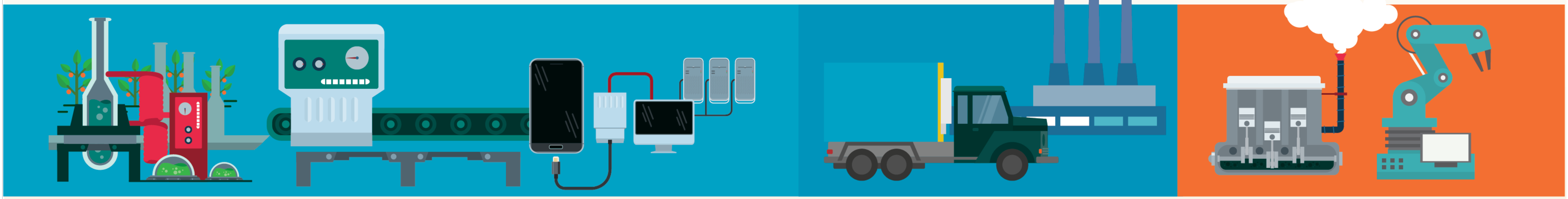

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*Enriching Engineering Science Through Collaboration of Multidisciplinary Fields*

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