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Preparation of Cobalt-Silica Dioxide (Co_xSiO_2) Catalyst through Impregnation Method

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ABSTRACT

The Co_xSiO_2 catalyst have been synthesized by reacting amount of cobalt nitrate and silica dioxide through impregnation method with vatiation on x= 0, 0.025, 0.05, and 0.1. The resulted materials were characterized using UV-Vis DRS, FT-IR, XRD, and SEM-EDX instruments. The XRD results showed that Co-SiO_2 catalyst have peaks at 2θ of 21.5º (for x = 0.025), 14.52º (for x = 0.05), and 21.58º (for x = 0.1). The Co_xSiO_2 catalysts were amorphous with average size of 4-11 nm and the band gap were at around of 1-3 eV.

Keyword: photodegradation, photocatalyst, methyl orange, Co_xSiO_2

1. INTRODUCTION

The industrial development lead to the increase of industry processes that directly causing the higher amount of industrial waste. Many researches have reported the negative effect of industrial waste to the environment (Nursabrina, 2021). Industrial waste produces toxic materials that harm the environment and human's life (Jadhav & Hocheng, 2012). According to the Ministry of Environment and Forestry in 2015 (KLHK, 2015), almost 68% of the water quality in Indonesian rivers were in heavily polluted status, especially by industrial waste.

Textile industry is one of the most developed industry, which mostly use toxic materials in the dyeing process, such as methylene blue, methyl orange, and congo red as commonly used azo dye which has a deterocyclic aromatic chemical compound that is difficult to decompose. Azo dyes have a chromophore system of azo group (-N=N) bonded to an aromatic group (Saranraj, et al, 2010). The reshuffle of azo dyes in water produces compounds that are more toxic and carcinogenic than the azo dyes themselves (Van Oudenhoven & Van Der Zee, 2002).

Several methods have been applied to handle the wasted dyes, including biological, physical, and chemical methods. Biological method, which usually called as bioremediation is being the most eco-friendly and less economical method. However, it has the disadvantage that it takes a long time to degrade. The adsorption method as one of the commonly used physical method, is relatively simple and quite effective to use, but this process only transfer the dye to the adsorbent. The chemical method has the advantage of rapidly degrade organic compounds into harmless products. One of the chemical methods used in waste handling is the photodegradation method using photocatalyst semiconductors (Widihati et al., 2011). Photocatalytic process will convert pollutants into more environmentally friendly products where the toxic and carcinogenic properties of pollutants will be degraded into harmless compounds (Kabra, 2004).

Silica is one of the photocatalysts that can be used to overcome water pollution. The photocatalytic activity of silica can be increased by engineering its surface chemistry. Metal doping is one of the surface modification method to increase the catalytic properties of silica (Decyk, et al, 2003). Cobalt has an outer electron configuration of 3d7 4s2 which can form complex compounds acting as Lewis acids, has widely used in the manufacture of doped-catalysts (Sibarani, 2012).

The selectivity and activity of the catalyst depend on the catalyst preparation method (Shimizu, et. al., 2009), which generally synthesized through sol-gel, co-precipitation and impregnation methods. The impregnation method is a catalyst preparation method in which the catalyst is prepared by attaching the active metal to a support material. The manufacturing process is simple, easy to carry out and affordable cost compared to sol-gel and co-precipitation methods, as well as better metal reproducibility (Pinna, 1998). In addition to the preparation method, the selection of the support material in the preparation of the catalyst also needs to be considered.

2. RESEARCH METHOD

Catalyst preparation using materials derived from SiO_2 and metallic cobalt (Co) to produce Co_xSiO2 compounds. With a variation of x = 0.025; x = 0.05; and x = 0.1. The impregnation preparation method was carried out by calcining the material at a temperature of 900ºC for 4 hours. The results of the preparation were then analyzed by XRD, FTIR, SEM-EDX and UV VIS DRS.

3. RESULTS AND ANALYSIS

Co_xSiO_2 catalyst has been successfully prepared is in the form of a fine gray powder which is getting clearer with the addition of cobalt concentration.

3.1 X-Ray Diffraction (XRD) Analysis

Catalyst characterization using X-Ray Diffraction (XRD) is a method used to identify the crystalline phase in a material by determining the lattice structure parameters. The XRD difactogram of the Co_xSiO_2 catalyst can be seen in Figure 1.

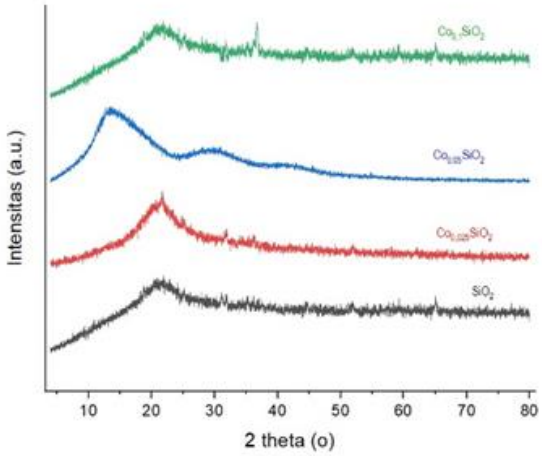


Figure 1. Diffractogram of Co_xSiO_2 at variation of Co concentration

Based on the diffraction pattern, it was found that the used SiO_2 compound is amorphous. The doping process with by calcined cobalt salt together with amorphous silica at 900oC for 4 hours produced the amorphous Co_xSiO_2 material. Figure 1 showed that the diffraction peaks pattern does not change significantly in the SiO_2, $Co_{0.025}SiO_2$ and $Co_{0.1}SiO_2$ samples where the characteristic amorphous SiO_2 with $2\theta = 21.08^º$ for x=0.025; $2\theta = 21.58^º$ for x=0.1 according to COD PDF number

76-1390. While the diffraction pattern for x=0.05 does not show a diffraction pattern that matches the amorphous SiO_2 reference, i.e. $2\theta = 14.52^{o}$ which will be discussed and proven in the FTIR and SEM-EDX characterization

3.2 FTIR Analysis

FTIR spectroscopy was used to identify and confirm the bonding type of the organic functional groups formed in the Co_xSiO_2 composite. The spectroscopic characterization of FTIR was carried out using the mid-infrared wavelength range as of 4000-400 cm^{-1} producing the spectra shown in Figure 2. The interpretation of FTIR spectra in Figure 2 are listed in Table 1.

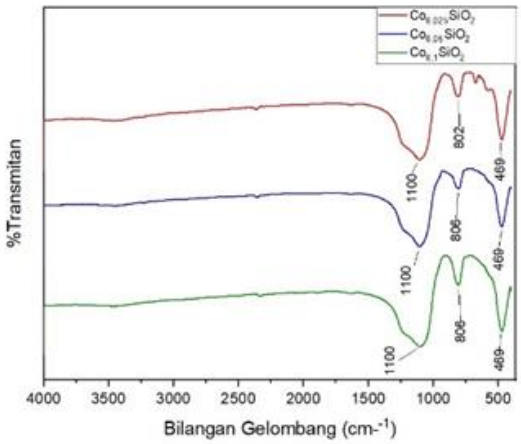


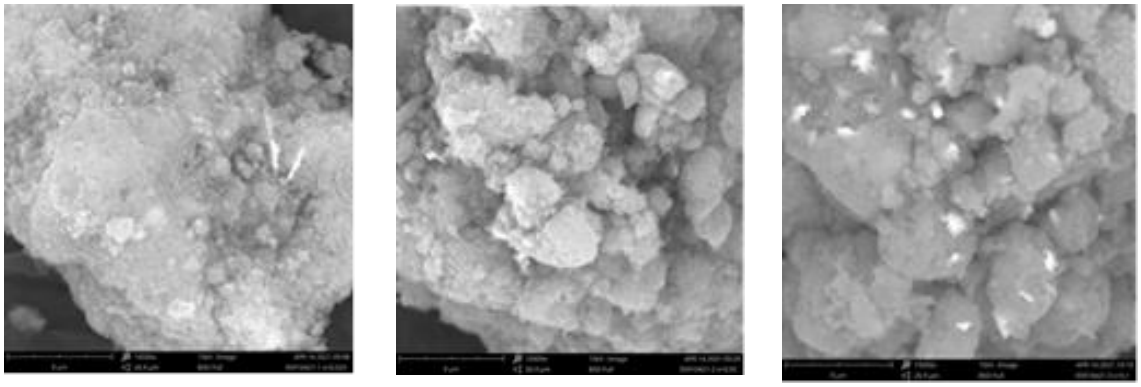
Figure 2. FTIR spectra of Co_xSiO_2

Table 1. The interpretation of Co_xSiO_2 spectra

Type of vibration	Wavenumber (cm-1)			
	Ref.	x = 0,025	x = 0,05	x = 0,1
Stretching of asymmetric Si-O-Si	1130-1000	1100	1100	1100
Stretching of Si-O	1100 -800	802	806	806
Bending of Si-O-Si	500-200	469	469	469

3.3 Scanning Electron Microscopy–Electron Dispersive X-ray (SEM-EDX) Analysis

SEM analysis with 10.000x magnificent are shown in Figure 3. Based on the results of SEM characterization in Figure 3A, it can be seen that the morphology of $Co_{0.025}SiO_2$ and $Co_{0.05}SiO_2$ in Figure 3B showed that the material surfaces are non-homogenous consisting consists of clumps (clusters) so that the particles look dominant in groups like giant silica structures. Clumping structure occurred because the SiO_2 amount was much higher compared to cobalt, so the surface morphology of the material tends to be like silica. The $Co_{0.1}SiO_2$ compound showed more homogenous grain size morphology in the sample. Morphological images from SEM in Figure 3 cannot explain the distribution of cobalt particles in the material structure, but the presence of cobalt is proven by the results of EDX analysis as listed in Table 2.



A B C

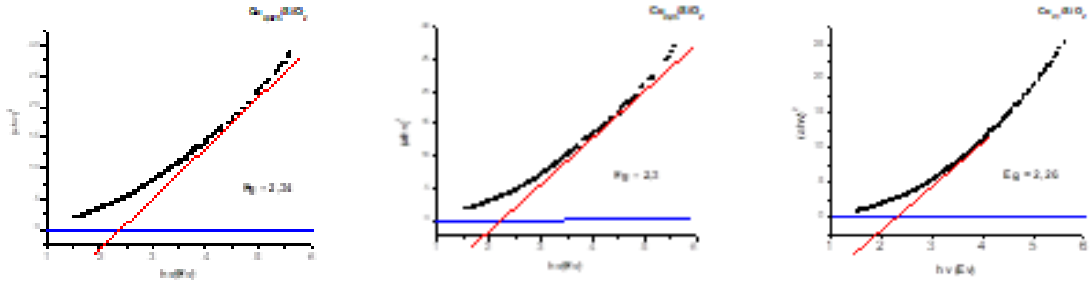
Figure 3. SEM analysis of Co_xSiO_2 with 10.000x magnificent (A = $Co_{0.025}SiO_2$; B = $Co_{0.05}SiO_2$; C = = $Co_{0.1}SiO_2$)

Table 2. EDX analysis of of Co_xSiO_2

Co_xSiO_2	Atomic ratio (%)		
	Co	Si	O
x = 0.025	0.66	23.85	47.90
x = 0.05	0.67	22.89	54.67
x = 0.1	1.72	27.76	63.41

3.4 UV-Vis Diffuse Reflectance (UV-Vis DRS) Analysis

UV-Vis DRS analysis was used to determine the band gap energy obtained from the wavelength produced using the Tauc Plot method as shown in Figure 4.



A B C

Figure 4. the Tauc Plot of Co_xSiO_2 (A = $Co_{0.025}SiO_2$; B = $Co_{0.05}SiO_2$; C = = $Co_{0.1}SiO_2$)

Based on the results of the analysis of the band gap energy values presented in Figure 4, it was found that $Co_{0.025}SiO_2$ has Eg of 2.34 eV, $Co_{0.05}SiO_2$ was 2.3 eV, and at $Co_{0.1}SiO_2$ was 2.26 eV. This indicates that the SiO_2 band gap energy value which was originally in the UV light region of 4.9 eV has shifted to the visible light region (1-3 eV) so that it is expected to be used in the photocatalyst process.

4. CONCLUSION

The Co_xSiO_2 catalyst was successfully prepared with various concentrations of cobalt. It gives a diffraction peak at 2θ = 21.08º for x = 0.025; 2θ = 14.52º for x = 0.05; and 2θ = 21.58º for x = 0.1. The Co_xSiO_2 catalysts has particle size between 4-11 nm with irregular morphology The resulting band gap energy ranges at around 2.3 eV.

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