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Nur Izzati Abu Bakar, University Technology Malaysia
Sheela Chandren, University Technology Malaysia
Nursyafreena Attan, University Technology Malaysia
Wai Loon Leaw, University Technology Malaysia
Hadi Nur, University Technology Malaysia

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Effect of magnetic field on the synthesis of well-aligned TiO$_2$-5CB by sol-gel method

Nur Izzati Abu Bakar $^{a,b}$, Sheela Chandren $^{b}$, Nursyafreena Attan $^{b}$, Leaw Wai Loon $^{a}$, Hadi Nur $^{a,c}$

$^a$ Centre for Sustainable Nanomaterials, Iban Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
$^b$ Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
$^c$ Central Laboratory of Minerals and Advanced Materials, Faculty of Mathematics and Natural Science, Universitas Negeri Malang (State University of Malang), Jl. Semarang 5 Malang 65145, Indonesia

Abstract

This paper describes the approach of using magnetic field as a technique to synthesize well-aligned materials. This magnetic field technique is method with high potential as the materials could be aligned by magnetic field as long as they possess magnetic anisotropy. The aim of this research is to explore the effects of magnetic field and magnetic line in the synthesis of well-aligned material, namely titania (TiO$_2$). The synthesis of well-aligned TiO$_2$ with liquid crystal as the structure-aligning agent is demonstrated under magnetic field in the presence of liquid crystal, 4'-pentyl-4-biphenylcarbonitrile (5CB), tetra-n-butyl orthotitanate (TBOT), 2-propanol and water. The mixture underwent slow hydrolysis and drying process under magnetic field (0.3 T) in ambient condition. The use of magnetic field and 5CB liquid crystal as the structure aligning agent has led to the successful formation of well-aligned TiO$_2$-5CB via sol-gel method. When no magnetic field was applied, the TiO$_2$-5CB obtained was spherical in shape and no alignment can be observed. This study demonstrated that magnetic field can play an important role in the synthesis of well-aligned TiO$_2$-5CB.

Keywords: Magnetic field; well-aligned; titania; sol-gel; slow hydrolysis

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in medical applications due to its biocompatibility [15]. Different shapes and sizes of TiO\textsubscript{2} have been reported to give different effects in various reactions such as photocatalytic reactions [15].

**EXPERIMENTAL**

**Materials**

The materials used in this research were 4-pentyl-4-biphenylcarbonitrile (5CB) (Sigma-Aldrich), which is a type of liquid crystal, tetra-n-butyl orthotitanate (TBOT) (Sigma-Aldrich) as the TiO\textsubscript{2} precursor, 2-propanol and distilled water.

**Synthesis of TiO\textsubscript{2}-5CB under magnetic field**

In a typical experiment, 4-pentyl-4-biphenylcarbonitrile (5CB) (0.023 mg), 2-propanol (2.057 ml) and distilled water (0.016 ml) were placed in a 5 ml sample bottle. TBOT (0.1 ml) was then added drop-wise into the mixture. Then, the solution was quickly transferred into a petri dish and covered with a perforated aluminium foil. The petri dish containing TBOT, 5CB, 2-propanol and distilled water was then placed under a magnetic field (0.3 T) and left to self-dry for 12 to 14 days. The magnetic strength applied was measured by a Handheld Gauss Meter teslameter. The relative humidity was 60%.

**Samples characterization**

Scanning electron microscopy (SEM) images on the synthesized TiO\textsubscript{2} were obtained using a JEOL JSM-6390LV instrument with an accelerating voltage of 15 kV. A small piece of the photocatalyst system is placed on a stub. In the instrument, a beam of highly energetic electrons was discharged towards the surface of the sample and the interaction between them fabricated signals, which were in the form of electrons. The SEM images produced can provide useful information on the surface morphology of the photocatalyst systems.

**RESULTS AND DISCUSSION**

**Formation of well-aligned TiO\textsubscript{2}-5CB**

Figure 1a and 1b shows the SEM images of TiO\textsubscript{2} sample synthesized in the presence of 5CB liquid crystal without magnetic field and under magnetic field, respectively. Well-aligned TiO\textsubscript{2}-5CB was successfully obtained when magnetic field was applied. On the contrary, when no magnetic field was applied, the TiO\textsubscript{2}-5CB formed was not aligned and the shape of TiO\textsubscript{2}-5CB was round-spherical shape. The difference between these two SEM images proves that the magnetic field affected the shape of TiO\textsubscript{2}-5CB. It is clearly observed that under magnetic field, the well-aligned TiO\textsubscript{2}-5CB was obtained.

![Figure 1](image)

**Factors affecting the formation of well-aligned TiO\textsubscript{2}-5CB**

In this study, a permanent magnet from neodymium block magnets was used as the source of magnetic field. Three neodymium block magnets were used and arranged according to the position of their north and south poles, as shown in Figure 2.

![Figure 2](image)

The arrangement of the neodymium block magnets, which affected the magnetic line of force, was taken into considerations. Thus, the effects of magnetic field lines towards different placement positions of the samples on the magnet bars were studied. In order to show the magnetic field lines on the magnet bar, iron powder, which is a ferromagnetic material, was used as an indicator. Figure 3c shows the placement of iron powder in the positions A and B, and the corresponding magnetic field lines pattern on the magnet bar (0.3 Tesla). It was obvious that the orientation and the density of the iron powder spread on the surface of the magnet bars were different. At position A, the strength of magnetic field is lower compared to the strength at position B. Figure 3d show the SEM images of TiO\textsubscript{2}-5CB synthesized under magnetic field at position A and B.

![Figure 3](image)

The SEM images of TiO\textsubscript{2}-5CB particles (Figures 3d) show interesting results, where the magnetic lines of force affected the morphology of the TiO\textsubscript{2}-5CB obtained. Distinctive effects on alignment of TiO\textsubscript{2}-5CB were vividly observed for the sample placed in the different position on the magnet bar. When the TBOT sample was placed at position A, the shape of TiO\textsubscript{2}-5CB obtained was “rambutan” like, whereas for TiO\textsubscript{2}-5CB synthesized at position B, the particles obtained were in the form of “skewers” like caused by the effect of magnetic line of force that occurred. This proves that the magnetic line of force affected the formation of TiO\textsubscript{2}-5CB, resulting in different morphologies.
Aside from magnetic field, it was found that the hydrolysis time also plays an important role in the formation of well-aligned TiO$_2$-5CB. As shown in Figures 4a and 4b, the effect of hydrolysis time for in the formation of well-aligned TiO$_2$-5CB synthesized under magnetic field can be clearly observed. Figure 4a shows the sample mixture containing 2-propanol with 0.08 v/v % water, while Figure 4b shows the sample mixture containing 2-propanol with 30 v/v % of water. Well-aligned TiO$_2$-5CB was obtained in the sample mixture that contains 0.08 v/v % water (Fig. 4a), while in mixture that contains 30 v/v % of water, the TiO$_2$-5CB was irregular in shape and not aligned (Fig. 4b).

Figure 4 Photographs and SEM images of sample mixtures that contain (a) 2-propanol with 0.08 v/v % of water and (b) 2-propanol with 30 v/v % of water.

Irregular-shaped TiO$_2$-5CB was obtained under relatively fast hydrolysis, while well-aligned TiO$_2$-5CB was formed under slow hydrolysis. It can be suggested that the formation of well-aligned TiO$_2$-5CB under magnetic field can only take place in slow hydrolysis condition. Thus, in order to ensure well-aligned TiO$_2$-5CB can be obtained, the amount of water in the reaction mixture needs to be controlled. The water content plays an important role to hydrolyse TBOT. If the water content used is too high, the TBOT will hydrolyse in too short of a time and the alignment of TiO$_2$ with 5CB liquid crystal under magnetic field cannot take place. This is because the amount of water will help speed up the hydrolysis process. On the contrary, when controlled amount of water is used, slow hydrolysis can occur, which then provide sufficient time for the 5CB to align TBOT under magnetic field. This alignment will not happen in fast hydrolysis, as there were insufficient time for the alignment to take place. Another important factor that should be highlighted in the formation of well-aligned TiO$_2$ is the good interfacial interaction between 5CB and TBOT. If the interfacial interaction is poor, the alignment would not have taken place.

CONCLUSION

In this study, well-aligned TiO$_2$-5CB has been successfully synthesized under magnetic field (0.3 T), by using 4'-pentyl-4-biphenylcarbonitride (5CB) liquid crystal as the alignment agent via the sol-gel method, coupled with a slow hydrolysis process. The liquid crystal was able to act as the alignment agent due to its magnetic properties and it was shown that the shape of TiO$_2$-5CB can be controlled by the induction of magnetic field, in the presence of 5CB liquid crystal. This new synthesis approach of utilizing magnetic field can be a novel way to design new photocatalyst.

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