

# Effect of magnetic field on the synthesis of well-aligned TiO<sub>2</sub>-5CB by sol-gel method

Nur Izzati Abu Bakar<sup>a,b</sup>, Sheela Chandren<sup>b</sup>, Nursyafreena Attan<sup>b</sup>, Leaw Wai Loon<sup>a</sup>, Hadi Nur<sup>a, c\*</sup>

<sup>a</sup> Centre for Sustainable Nanomaterials, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

<sup>b</sup> Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

<sup>c</sup> Central Laboratory of Minerals and Advanced Materials, Faculty of Mathematic and Natural Science, Universitas Negeri Malang (State University of Malang), Jl. Semarang 5 Malang 65145, Indonesia

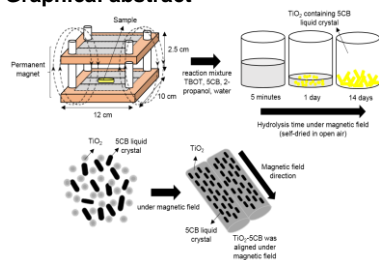
\* Corresponding author: hadi@kimia.fs.utm.my

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## Graphical abstract



## 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<sub>2</sub>). The synthesis of well-aligned TiO<sub>2</sub> 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<sub>2</sub>-5CB via sol-gel method. When no magnetic field was applied, the TiO<sub>2</sub>-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<sub>2</sub>-5CB.

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

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## INTRODUCTION

Magnetic field is a mathematical description of the magnetic influence towards magnetic materials. It is produced by moving electric charges and the intrinsic magnetic moments of elementary particles associated with a fundamental quantum property, which is their spin [1-3]. In everyday life, magnetic fields are most often encountered as an invisible force created by permanent magnets that pulls on iron objects and attracts or repels other magnets [4]. Permanent magnets are objects that are able to produce their own persistent magnetic fields. These magnets are often made of ferromagnetic materials, such as iron and nickel, that have been magnetized, and they have both a north and a south pole [5].

Most materials respond to magnetic field by producing their own magnetism. This magnetism is one of the main physical properties of materials and every material possesses its own magnetism [5-6]. Magnetism can be classified into three groups, which are ferromagnetism, paramagnetism and diamagnetism. For example, iron and nickel are substances that possess ferromagnetic properties. Paramagnetic materials are materials that are attracted to magnetic fields, such as in the case of oxygen gas. Diamagnetic materials are materials that are unaffected by magnetic fields. Water is included in this category [5-6]. These magnetic properties are due to the magnetic susceptibility or magnetic energy density of the material [5]. Therefore, it is expected that magnetic field will affect the physical properties, which can be used to control chemical and physical processes.

The effects of magnetic fields are categorized into four types; magneto-thermodynamic effect, quantum effect, magnetic force, and magnetic torque and alignment [7-10]. From 1992 to 1994, the effects of magnetic field in controlling chemical and physical processes have been extensively studied in Japan, as the importance of high magnetic field has been recognized [7]. In a research project entitled "Innovative Utilization of High Magnetic Field", it was stated that many interesting phenomena, which can hardly be detected in low magnetic field, have been observed. It should be highlighted that these chemical and physical processes can be associated even with diamagnetic materials [7]. For example, magnetic orientation of organic polymers, gels and carbon nanotubes, magnetic levitation of diamagnetic materials, and pseudo-microgravity generated by magnetic force have all been observed [11-13]. These newly found magnetic phenomena will be very useful for processing functional molecules with improved quality.

Therefore, in this research, the magnetic field technique shows high potential as all materials, even diamagnetic materials, can be aligned by magnetic field as long as they possess magnetic anisotropy. Magnetic anisotropy is also known as the material's magnetic properties. The material that has been utilized in this research was tetra-*n*-butyl orthotitanate (TBOT) which is a precursor of titania (TiO<sub>2</sub>). TiO<sub>2</sub> is currently the most important, most widespread and most investigated metal oxide due to its low toxicity, high thermal stability, and broad applicability [14]. With its semiconductor properties, TiO<sub>2</sub> has shown outstanding performance in photocatalysis, water-splitting and self-cleaning [14]. It is also useful

in medical applications due to its biocompatibility [15]. Different shapes and sizes of  $\text{TiO}_2$  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  $\text{TiO}_2$  precursor, 2-propanol and distilled water.

### Synthesis of $\text{TiO}_2$ -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 dropwise 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%.

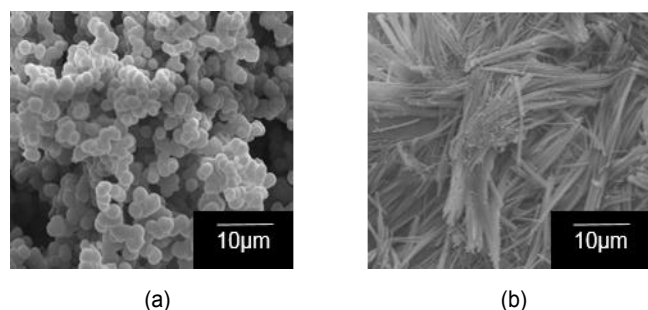
### Samples characterization

Scanning electron microscopy (SEM) images on the synthesized  $\text{TiO}_2$  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 $\text{TiO}_2$ -5CB

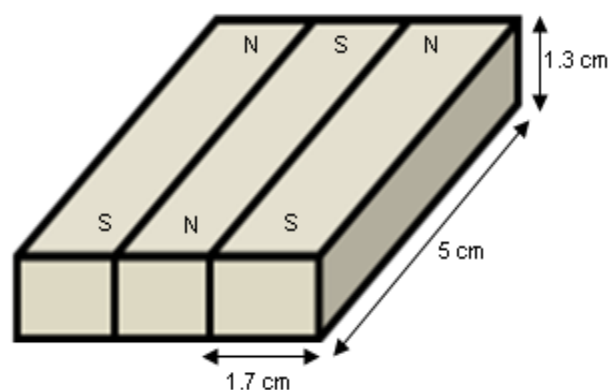
Figure 1a and 1b shows the SEM images of  $\text{TiO}_2$  sample synthesized in the presence of 5CB liquid crystal without magnetic field and under magnetic field, respectively. Well-aligned  $\text{TiO}_2$ -5CB was successfully obtained when magnetic field was applied. On the contrary, when no magnetic field was applied, the  $\text{TiO}_2$ -5CB formed was not aligned and the shape of  $\text{TiO}_2$ -5CB was round-spherical shape. The difference between these two SEM images proves that the magnetic field affected the shape of  $\text{TiO}_2$ -5CB. It is clearly observed that under magnetic field, the well-aligned  $\text{TiO}_2$ -5CB was obtained.



**Figure 1** SEM images of  $\text{TiO}_2$  sample synthesized in the presence of 5CB liquid crystal a) without magnetic field, b) under magnetic field.

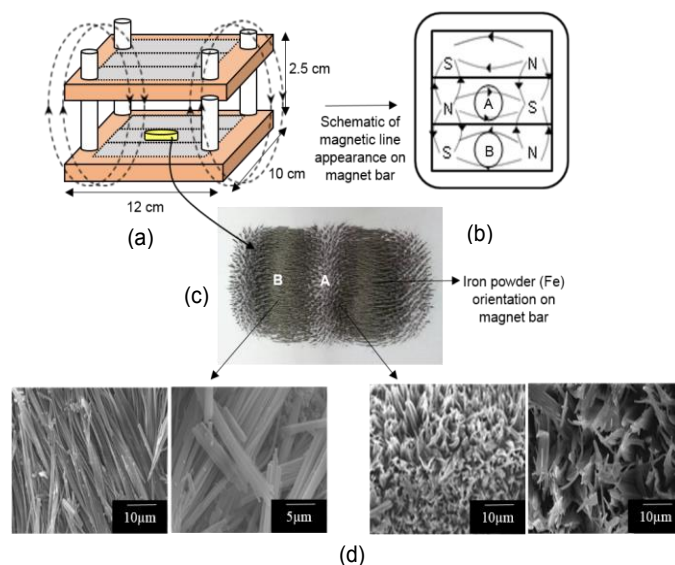
### Factors affecting the formation of well-aligned $\text{TiO}_2$ -5CB

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** The arrangement of neodymium block magnets.

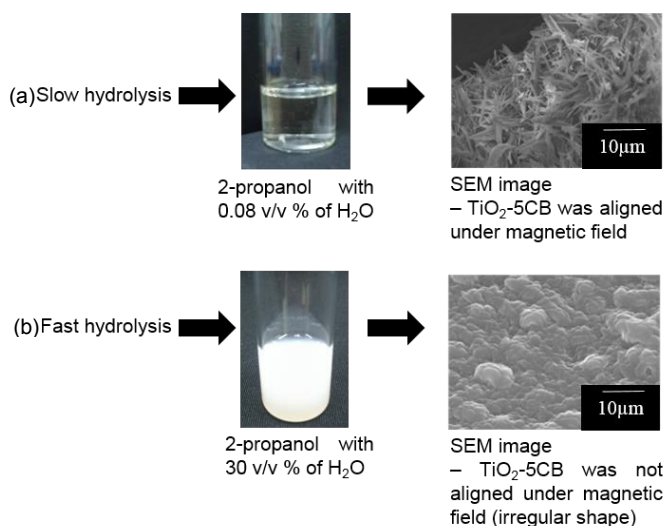
The arrangement of the neodymium block magnets, which affected the magnetic line of force, was taken into considerations. Thus, the effects of magnetic field lines towards different placement positions of the samples on the magnet bars were studied. In order to show the magnetic field lines on the magnet 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 shows the SEM images of  $\text{TiO}_2$ -5CB synthesized under magnetic field at position A and B.



**Figure 3** (a) and (b) Schematic diagram of magnetic line appearance on magnet bar, (c) iron powder (Fe) orientation on magnet bar and (d) SEM images of  $\text{TiO}_2$ -5CB particles synthesized at position A and B on the magnet bar.

The SEM images of  $\text{TiO}_2$ -5CB particles (Figures 3d) show interesting results, where the magnetic lines of force affected the morphology of the  $\text{TiO}_2$ -5CB obtained. Distinctive effects on alignment of  $\text{TiO}_2$ -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  $\text{TiO}_2$ -5CB obtained was “rambutan” like, whereas for  $\text{TiO}_2$ -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  $\text{TiO}_2$ -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  $\text{TiO}_2$ -5CB. As shown in Figures 4a and 4b, the effect of hydrolysis time for in the formation of well-aligned  $\text{TiO}_2$ -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  $\text{TiO}_2$ -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  $\text{TiO}_2$ -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  $\text{TiO}_2$ -5CB was obtained under relatively fast hydrolysis, while well-aligned  $\text{TiO}_2$ -5CB was formed under slow hydrolysis. It can be suggested that the formation of well-aligned  $\text{TiO}_2$ -5CB under magnetic field can only take place in slow hydrolysis condition. Thus, in order to ensure well-aligned  $\text{TiO}_2$ -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  $\text{TiO}_2$  with 5CB liquid crystal under magnetic field cannot take place. This is because the amount of water will help speed up of 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  $\text{TiO}_2$  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  $\text{TiO}_2$ -5CB has been successfully synthesized under magnetic field (0.3 T), by using 4'-pentyl-4-biphenylcarbonitrile (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  $\text{TiO}_2$ -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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