



Fabrication of Fiber Optic Magnetic Field Sensors with Ferrofluid Fe₃O₄/DMSO Synthesized Using the Laser Ablation Method

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Ila Nur Alifah Islami¹, Nurul Hidayat¹, Muhammad Safwan Abd Aziz², Arif Hidayat¹, Lya Rizka Herawati¹, ST. Ulfawanti Intan Subadra¹, Ahmad Taufiq^{1*}

1. Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Malang, Jl. Semarang 5, Malang, 65115, Indonesia
2. Laser Center, Ibnu Sina Institute for Scientific & Industrial Research (ISI-SIR), Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia

*Corresponding Author's E-mail: ahmad.taufiq.fmipa@um.ac.id



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Abstract

Currently, the demand for practical, highly sensitive magnetic field sensors is increasing in the industrial and medical sectors. Optical fiber magnetic field sensors are an innovation that can provide a solution to this demand. One development in optical fiber magnetic field sensors involves modifying the ferrofluid material used as the sensing material. In this study, Fe₃O₄/DMSO ferrofluid was synthesized using the laser ablation method. The synthesis results were then integrated with SNS (SMF – NCF – SMF) structured optical fibers to form a magnetic field sensor. The XRD results showed amorphous samples originating from glass as the Fe₃O₄/DMSO substrate. The FTIR spectrum confirmed the presence of Fe₃O₄ with the detection of octahedral and tetrahedral Fe-O functional groups. PSA characterization showed a particle size distribution of 343 nm. Furthermore, the UV-Vis spectrum absorbance showed a characteristic peak of iron oxide nanoparticles, namely at an absorption peak of 341.7 nm, and had a band gap of 2.04 eV. The performance results of the optical fiber magnetic field sensor using Fe₃O₄/DMSO ferrofluid show that the sensor is sensitive with a sensitivity of $1.2319e^{-5} \pm 1.23477e^{-6}$ a.u./mT with a linear response $R^2 = 0.9431$.

Keywords: Ferrofluid, Fe₃O₄/DMSO, Laser Ablation, Magnetic Field Sensor, Optical Fiber

1. Introduction

Magnetic field sensors are devices commonly found in various industrial [1] and medical [2]. technologies. Advancements in technology demand more sophisticated, flexible, and practical magnetic field sensors. The emergence of fiber optic-based sensors with a simple design addresses the need for innovative magnetic field sensors with superior performance. The advantage of using fiber optics as sensors is that they are easy to fabricate with a simple structure and a small size. This makes fiber optic-based magnetic field sensors easier to reach narrow detection positions [3].

Optical fiber-based magnetic field sensors use optical fibers as the main material in manufacturing sensors by utilizing perfect internal reflection mode. The optical fibers used are usually modified so that internal reflection can reach the magnetic field detection area. Therefore, the manufacture of optical fiber magnetic field sensors requires sensing materials that respond to changes in the magnetic field [4]. Yan et al. developed a single mode fiber – no core fiber – single mode fiber (SNS) optical fiber structure coated with graphene oxide (GO). This sensor exhibits a magnetic field sensitivity of 0.015 – 0.043 nm/mT in the range of 0–18 mT[5]. In another study, Li et al. reported the use of single-mode fiber (SMF) integrated with Fe₃O₄/OA on polydimethylsiloxane (PDMS) to detect magnetic field strength in the range of 0 – 200 mT with a sensitivity of -62.8 pm/mT [6].

Based on previous studies, sensor performance is highly dependent on the fiber structure and sensing material used. Sensing materials require materials that are responsive when interacting with external magnetic fields. Ferrofluid is one such responsive material consisting of nano- to micron-sized magnetic particles dispersed in a liquid [7]. Magnetic particles in liquids interact more easily and orient themselves toward external magnetic fields, thereby increasing the sensitivity and responsiveness of the sensor. Magnetic particles in ferrofluid usually consist of ferrite particles, such as magnetite Fe_3O_4 [8], $\gamma\text{-Fe}_2\text{O}_3$ [9], NiFe_2O_4 [10], or metal particles such as cobalt (Co), nickel (Ni), iron (Fe), and their alloys [11]. Li *et al.*, have shown that the use of $\text{Fe}_3\text{O}_4/\text{OA}$ ferrofluid can produce good performance [6]. Therefore, in this study, an optical fiber magnetic field sensor will be made using Fe_3O_4 as the sensing material.

The synthesis of Fe_3O_4 ferrofluid has been widely carried out using several methods such as coprecipitation, precipitation, hydrothermal, sol-gel, microemulsion, and sonochemistry [7], [12]. Saputro *et al.* (2020) successfully synthesized $\text{Fe}_3\text{O}_4/\text{OA}/\text{DMSO}$ ferrofluid using the sonochemistry method [13]. However, this method is ineffective because it uses many hazardous chemicals and the fabrication process is long and complicated [14]. Therefore, this study used a simpler synthesis method using the laser ablation synthesis technique.

Laser ablation synthesis is a top-down synthesis technique using lasers that is easy, fast, and environmentally friendly. Nanoparticle synthesis using the laser ablation method is carried out by focusing a laser beam on bulk material in a liquid medium [15]. This technique minimizes the use of additional hazardous chemicals and can produce high-purity materials because the process uses pure raw materials, which minimizes impurities. Furthermore, this study used DMSO as a surfactant that can reduce Van der Waals forces by interacting with materials in liquids [14], so that the resulting ferrofluid is more stable and has superior magnetic properties as an effective magnetic field sensor.

2. Methods

Synthesis of $\text{Fe}_3\text{O}_4/\text{DMSO}$ Ferrofluid Material Using the Laser Ablation Method

In this study, Fe_3O_4 ferrofluid was synthesized using Fe bulk (99.99%), dimethyl sulfoxide (DMSO), borges olive oil (99.92%), and distilled water. The synthesis process of Fe_3O_4 ferrofluid was carried out using the laser ablation method. The instrument used in this method was a Fiber Laser Marking Machine with the help of EZCAD software. Fe bulk was placed in a dish containing distilled water, then the laser was focused on the bulk Fe for 5 minutes. The laser was fired at a power of 10.28 mJ with a wavelength of 1064 nm and a laser firing frequency of approximately 100 shots [16]. During this process, Fe_3O_4 nanoparticles are formed and dispersed in the liquid. After that, 2 ml of DMSO is dissolved in 10 ml of olive oil and stirred for 1 hour at a temperature of 90 °C [13]. Three milliliters of this solution is taken and added to the Fe_3O_4 nanoparticles as a surfactant. The sample was then tested using Fourier Transform Infrared Spectroscopy (FTIR), Particle Size Analyzer (PSA), Ultraviolet-Visible Spectrophotometer (UV-Vis), and optical fiber magnetic field sensor testing. Next, the sample was dried using the dropcasting method on a glass film for X-ray Diffraction (XRD) testing.

Fabrication and Testing of Optical Fiber Magnetic Field Sensors with $\text{Fe}_3\text{O}_4/\text{DMSO}$ Ferrofluid

The optical fiber magnetic field sensor was fabricated using Single Mode Fiber (SMF) and No Core Fiber (NCF) optical fibers with an SNS (SMF – NCF – SMF) structure. The SMF used had a length of 7 cm, while the NCF used had a length of 1.5 cm. Next, the spliced optical fibers were inserted into a capillary tube filled with Fe_3O_4 ferrofluid. Finally, both ends of the capillary tube were sealed using glue. The shape of the optical fiber magnetic field sensor is shown in Figure 1. The sensitivity of the optical fiber magnetic field sensor was tested by connecting one end of the optical fiber to a light source and the other end to a detector. The spectrum of changes in the strength of the magnetic field sensor in response to light can be viewed using the Thorlabs application.

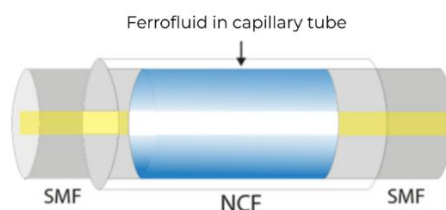


Figure 1. Structure of the Optical Fiber Magnetic Field Sensor.

3. Results and Discussion

The XRD characterization results of $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid are shown in Figure 2. In general, the crystallization of $\text{Fe}_3\text{O}_4/\text{DMSO}$ is indicated by diffraction peaks at $2\theta = 30^\circ, 35^\circ, 44^\circ, 54^\circ, 57^\circ, 64^\circ,$ and 75° [17]. However, in this study, these peaks did not form, and the characterization results showed amorphous peaks at $2\theta = 20^\circ\text{--}30^\circ$. These peaks indicate the diffraction of the glass used as a substrate for drying $\text{Fe}_3\text{O}_4/\text{DMSO}$. This is because the low concentration of Fe_3O_4 material compared to the surfactant and liquid on the substrate makes the diffraction peak invisible, so that the material detected by the XRD shows a diffraction pattern of amorphous glass (silica). This is also shown in the study conducted by Falahudin [18], [19].

The FTIR spectrum of $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid is shown in Figure 3. The Fe_3O_4 material was confirmed by the detection of octahedral Fe-O bond vibrations at 503 cm^{-1} and tetrahedral Fe-O vibrations at 601.8 cm^{-1} and 833 cm^{-1} [20][21][22]. The addition of DMSO surfactant was identified at a wavenumber of 1103.3 cm^{-1} , which is an S=O functional group, and the presence of CH_2 functional groups at 1473 cm^{-1} and 2858 cm^{-1} [23][24]. In addition, peaks were also observed at wave numbers 1280 cm^{-1} and 1236 cm^{-1} which identified the CH_3 functional group, confirming the presence of DMSO surfactant [25]. The presence of olive oil as a solvent medium in $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid is also confirmed by the presence of C-H bonds at wave numbers 2962 cm^{-1} and 2935 cm^{-1} [13]. The peak at 2360 cm^{-1} indicates CO_2 bonds originating from air trapped in the sample [26]. Finally, the broad band around the wavenumber 3568 cm^{-1} indicates the presence of O-H stretching vibrations [16][27].

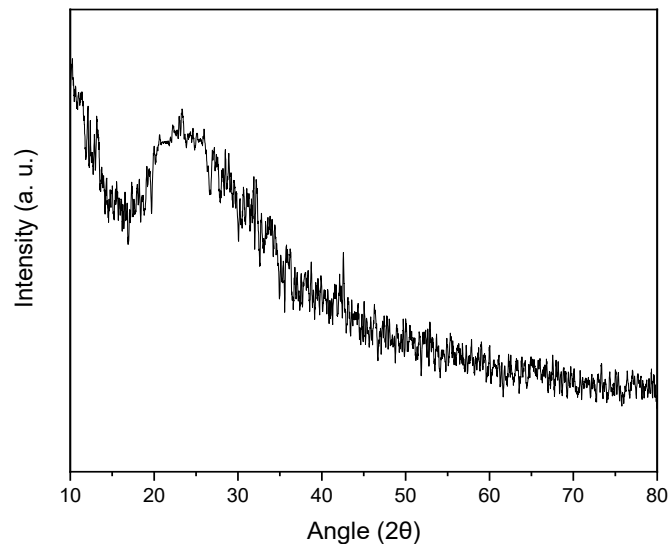


Figure 2. XRD Pattern of $\text{Fe}_3\text{O}_4/\text{DMSO}$.

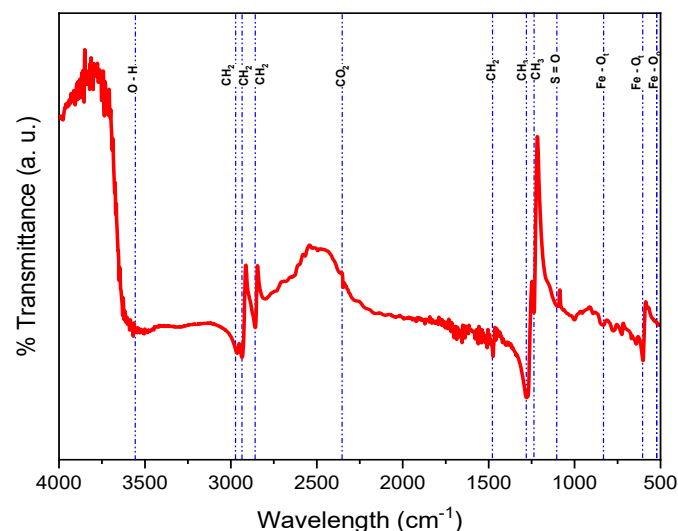


Figure 3. FTIR Spectrum of $\text{Fe}_3\text{O}_4/\text{DMSO}$ Ferrofluid.

Particle size was determined based on PSA characterization using the dynamic light scattering (DLS) method. The average particle size distribution in the PSA characterization results can be seen in Figure 4, which shows that the $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid has an average particle size of approximately 343 nm. In the same study by Koubanani *et al.*, there were differences in the measurement results using TEM and PSA characterization, with average particle sizes of 5–30 nm and 284 nm, respectively [28]. This is due to the DLS method being based on the dominance of light scattering by agglomerated particles, so that smaller free particles are covered and cannot be detected [29]. Furthermore, the agglomeration that occurred in this study was caused by magnetic dipole interactions and van der Waals forces when $\text{Fe}_3\text{O}_4/\text{DMSO}$ mixed with distilled water [30].

The UV-Vis absorption spectrum of $\text{Fe}_3\text{O}_4/\text{DMSO}$ material is shown in Figure 5. $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid has an absorption band in the interval of 285–590 nm with an absorption peak at 341.7 nm, which is a typical peak of iron oxide nanoparticles [31]. The width of the absorption peak may be caused by particle agglomeration [32]. Furthermore, the bandgap value of the $\text{Fe}_3\text{O}_4/\text{DMSO}$ ferrofluid is 2.04 eV. This result is close to the bandgap value of Fe_3O_4 nanoparticles reported by Attallah *et al.* The absorption peak and bandgap values confirm that this study has successfully produced Fe_3O_4 [33].

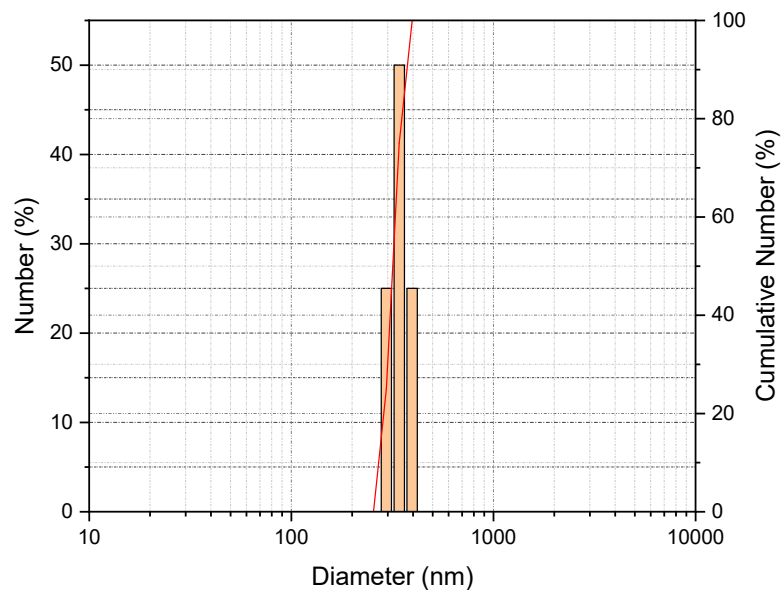


Figure 4. Particle Size Analyzer Spectrum of Ferrofluid $\text{Fe}_3\text{O}_4/\text{DMSO}$.

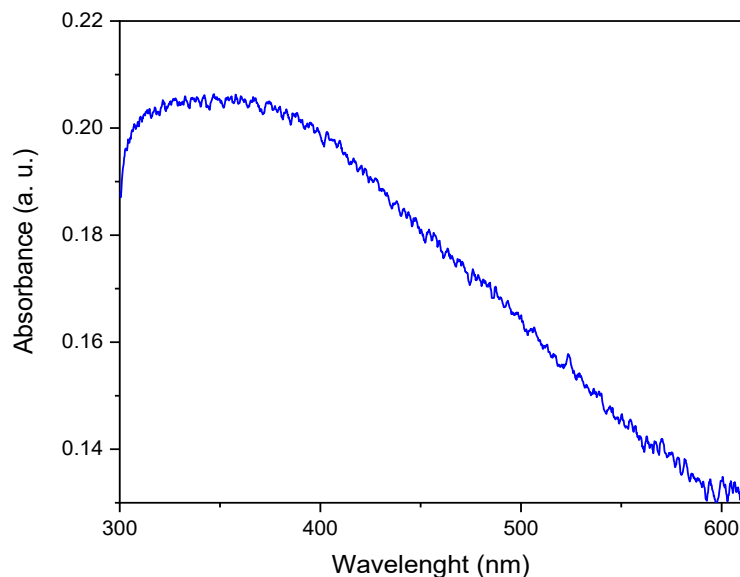


Figure 5. UV-Vis of $\text{Fe}_3\text{O}_4/\text{DMSO}$ Ferrofluid.

The performance results of the optical fiber magnetic field sensor using Fe₃O₄/DMSO ferrofluid are shown in Figure 6 (a). The phenomenon of magnetic particle distribution caused by changes in the external magnetic field will affect the refractive index in Fe₃O₄/DMSO ferrofluid. Changes in light intensity are related to changes in the refractive index caused by the movement of Fe₃O₄ material due to the external magnetic field. Therefore, in this study, the light intensity will change along with changes in the external magnetic field [34]. An increase in the strength of the external magnetic field is proportional to the change in transmitted light intensity. The external magnetic field can penetrate the surfactant so that the Fe₃O₄ nanoparticles inside it can interact and cause changes in intensity in the sensor [35].

In Figure 6 (b), the value of $R^2 = 0.9431$ (degree of linearity) shows the linearity of the sensor response, which supports the sensor sensitivity results in the measurement range of 100–240 mT. These results indicate the stability of the Fe₃O₄/DMSO ferrofluid material as a sensing material in the range of 100–240 mT. Based on the slope graph, the magnetic field sensor sensitivity is $1.2319 \times 10^{-5} \pm 1.23477 \times 10^{-6}$ a.u./mT. This study shows that the sensor remains stable and can detect changes in the external magnetic field up to 240 mT. This is an improvement in the magnetic field strength detection range compared to previous research conducted by Revathi et al., with a maximum range of ~190 mT [36].

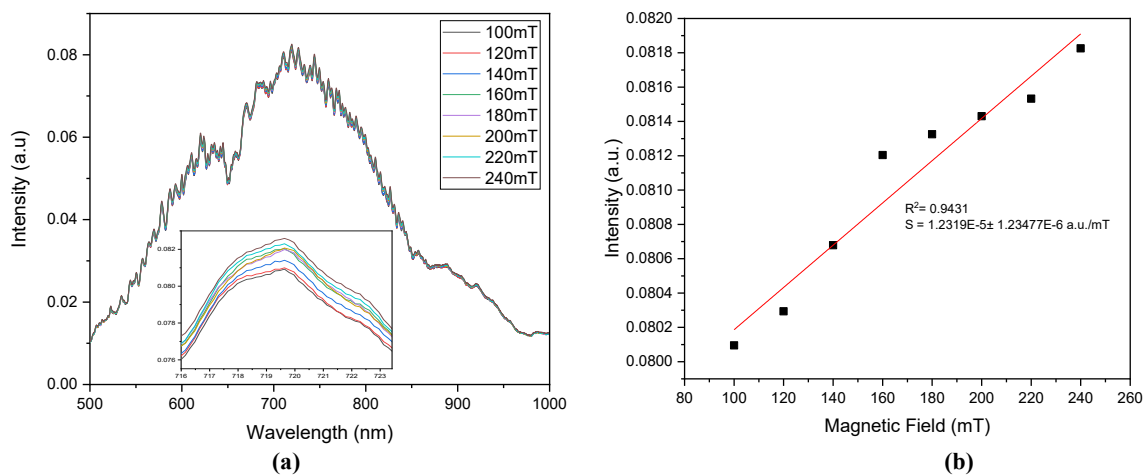


Figure 6. (a) Spectrum Response and (b) Sensitivity of Fe₃O₄/DMSO Ferrofluid to Magnetic Field.

4. Conclusion

Fe₃O₄/DMSO ferrofluid has been successfully synthesized using the laser ablation method. The results show that the XRD pattern of Fe₃O₄/DMSO ferrofluid is dominated by the amorphous diffraction pattern of the glass substrate. The Fe₃O₄/DMSO material was confirmed by FTIR and UV-Vis spectroscopy, where the FTIR results detected octahedral and tetrahedral Fe-O functional groups which indicating the presence of Fe₃O₄, then S=O, CH₂, and CH₃ functional groups which indicating the presence of DMSO, and the UV Vis results produced an absorption peak at 341.7 nm and a band gap of 2.04 eV. Furthermore, the PSA results showed an average particle size of 343 nm. The performance results of the optical fiber magnetic field sensor using Fe₃O₄ DMSO ferrofluid showed a sensitivity of $1.2319 \times 10^{-5} \pm 1.23477 \times 10^{-6}$ a.u./mT with a detection range of 100–240 mT. Thus, the optical fiber magnetic field sensor using Fe₃O₄/DMSO ferrofluid can be considered a practical and sensitive sensor.

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