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DETERMINING THE PARTICLE SIZE OF TiO_2/EG-WATER XRD DATA USING THE SCHERRER EQUATION

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ABSTRACT

X-ray diffraction (XRD) data and the Scherrer equation were utilized to analyze the particle size of titanium dioxide (TiO_2) in a solution of ethylene glycol (EG) and distilled water. The XRD analysis was conducted using a Rigaku Miniflex 600 instrument with an X-ray wavelength of approximately 0.15046 nm. The examination yielded the full-width half maximum (FWHM), which was subsequently examined using the Scherrer equation. This experiment employed TiO_2 with a purity level of 99.8% and a particle size of 30 nm. The analysis revealed that the average particle size of TiO_2 in the sample is 19.45 nm, with the highest measurement at about 30.38 nm. The Spearman correlation equation was employed to validate the outcomes. The Spearman's correlation coefficient between the FWHM variable and the particle size of TiO_2 nanoparticles is -0.958. These findings shed light on the particle structure of TiO_2 under these conditions. These findings lend support to the use of TiO_2 in a variety of nanotechnology applications. However, more research is needed to understand how particle-size TiO_2 nanoparticles work in different settings and to find the best ways to prepare samples, including understanding the specific phase and how it affects the stability of fluids. This research contributes significantly to the understanding of the properties of TiO_2 in a solution of distilled water and EG, as well as to the characterization of nanomaterials, with particular emphasis on issue 9 of the SDGS Goal concerning industry, innovation, and infrastructure.

Keywords: Particle size; X-ray Diffraction; Ethylene Glycol; Scherrer Equation; TiO_2 nanoparticle.

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INTRODUCTION

Nanotechnology is an area of research rapidly developing and causing significant changes in many fields of science and technology. Nanomaterials have distinct particle structures and small sizes of particles that are typically less than 100 nm [1]. Furthermore, determining the minuscule dimensions of nanomaterials frequently presents a formidable barrier. A common obstacle to the application of Scanning Electron Microscopy (SEM) and

Transmission Electron Microscopy (TEM) imaging is the phenomenon of agglomeration. Meanwhile, nanomaterials also offer extraordinary properties and applications. In this

context, Titanium Dioxide (TiO_2) has become a nanomaterial that has attracted significant attention from researchers and industry [2]. Several applications of TiO_2 as TiO_2/EG-water nanofluid are presented in Figure 1 [3].

TiO_2 is one of the most widely used nanomaterials and has a variety of essential applications [4], including in photocatalysis, photovoltaics, optoelectronics, and many more [5]. The particle size or particlelinity of TiO_2 is significant in determining its properties and performance in this application. For this reason, accurate analysis regarding the size of TiO_2 particles has become a significant focus of nanomaterial research [6].

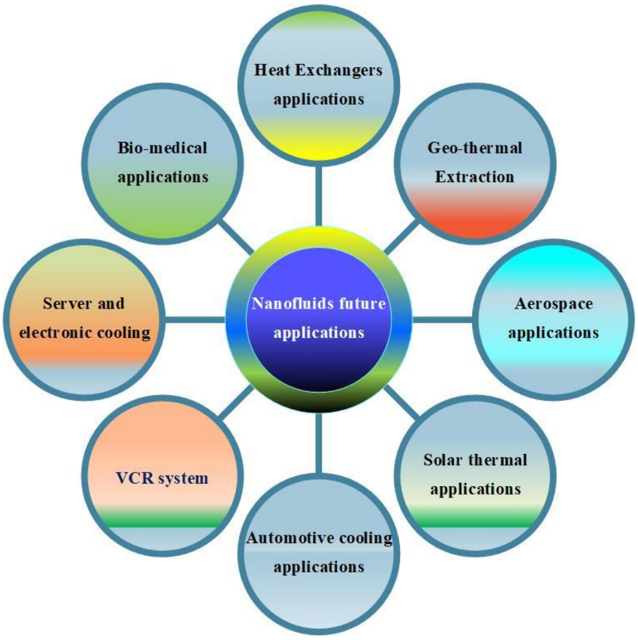

Figure 1. Potential future applications of nanofluids [3].

X-ray Diffraction (XRD) is one of the most commonly used techniques for characterizing the particle structure of nanomaterials [7]. The Scherrer equation is one of the most critical methods in XRD data analysis. The Scherrer equation is a powerful tool for calculating the size of particles (particles) based on the width of the diffraction peaks in XRD data [8-9].

Several studies have been carried out on the characterization of the particle structure of nanomaterials. Research using the XRD pattern method using the Debye Scherer formula was carried out by [10] using the peak-broadening analysis method with TiO_2 nanoparticle material measuring 1-0.7 micrometers. Furthermore, research on Particle size using TiO_2/water was carried out by [11]. The TiO_2 nanoparticles used have a size of 30 nm. Analysis was done using the reference code JCPDS-211272 and a quadratic particlelization system. The resulting research results show data higher than the specifications with a particle size in the range of 33.46606A- 73.64458A. It has converted around 3.4-7.4 nm. In other research, [12] researched TiO_2 nanoparticles synthesized from natural materials with a nanoparticle size of around 31–42 nm.

This study employs the Scherrer Equation analysis technique to determine the particle size of TiO_2 nanomaterial dispersed in a mixture of ethylene glycol (EG) and distilled water. The main goal is to determine the size of TiO_2 particles using an XRD test data analysis method that utilizes the Scherrer equation. Therefore, it is essential to dry out the nanofluid to improve the accuracy of the XRD test results. The TiO_2 nanoparticles utilized in this study

have a size of approximately 30 nm, which is smaller than the nanoparticles used in previous research. The size of particles within these nanomaterials is determined utilizing a dependable and precise methodology. The Spearman correlation is utilized to validate the results of the calculations, providing possible improvements in the research and development of TiO_2-based nanomaterials for diverse technological applications.

METHOD

Material of TiO_2/EG-water Nanofluid

This study used TiO_2 nanoparticles with batch code 20220817 from Hebei Suoyi New Material and Technology Co., Ltd. These nanoparticles have a purity level of around 99.8% and have a rutile particle structure. The raw material for TiO_2 nanoparticles is solid powder, which has a white physical appearance, and the average specific size is around 30 nm. The primary fluids used in this research were EG and water. An overview of the TiO_2 nanoparticles, EG, and water used can be seen in Figure 2.

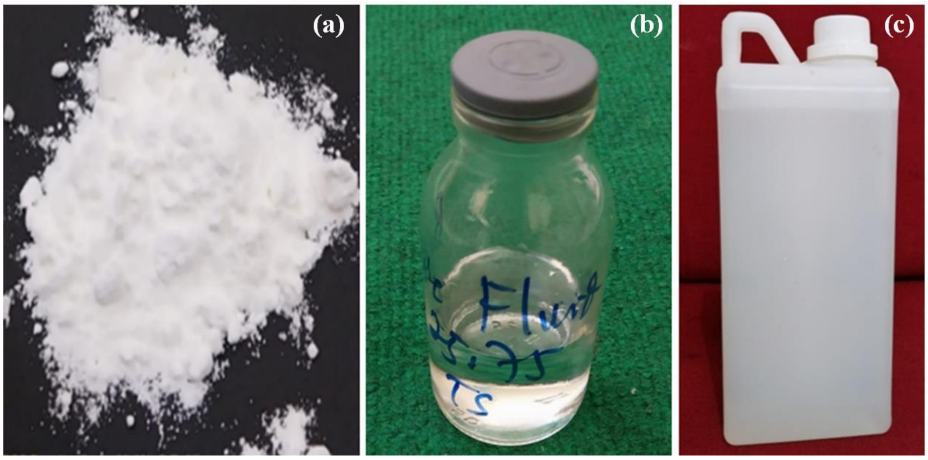

Figure 2. Raw material: (a) TiO_2-Powder, (b) ethylene glycol (EG) and (c) water

Preparation of TiO_2/EG-water Nanofluid

The TiO_2 nanoparticle preparation process was carried out using the two-step approach [13]. The two-step method is a method that has been studied in the literature [14-15] TiO_2 nanoparticles in powder form are dispersed into a base liquid to form nanofluids [16]. This process involves mixing the nanoparticle powder with the base fluid and stirring to achieve homogeneous dispersion of the nanoparticles in the base fluid [17].

In this research, TiO_2 powder nanoparticles were with a volume fraction of 1.75 dispersed in EG/ water fluid with a volume percentage of 25:75. The final result of this mixing will form a mixture of TiO2/EG-water. It refers to the percentage of EG concentration used at about 25%. The first step in creating a TiO_2/EG-water nanofluid with a volume fraction of 1.75% is to prepare the necessary raw materials. This method employs TiO_2 nanoparticles that have been precisely tested and measured to the desired size and EG as a base fluid. Figure 3 provides the two-step method approach used in this study.

The subsequent step involves an intensive mixing process to ensure the even distribution of TiO_2 particles within the base liquid. Ultrasonication methods can further enhance dispersion and prevent particle agglomeration [18]. Subsequently, a meticulous quality assessment of the produced nanofluid is imperative through appropriate analyses, which encompass evaluating particle size, particle size distribution, and suspension stability [5].

Suppose the volume fraction of TiO_2 in the nanofluid falls short of 1.75%. In that case, adjustments can be made by incrementally introducing more TiO_2 as needed, emphasizing thorough mixing with each addition. Once the nanofluid attains the desired volume fraction, it should be securely stored in a suitable container to forestall contamination. It is crucial to bear in mind that rigorous control over both conditions and composition constitutes an indispensable aspect of nanofluid manufacturing, and comprehensive characterization of nanofluids following the objectives of their intended application is paramount.

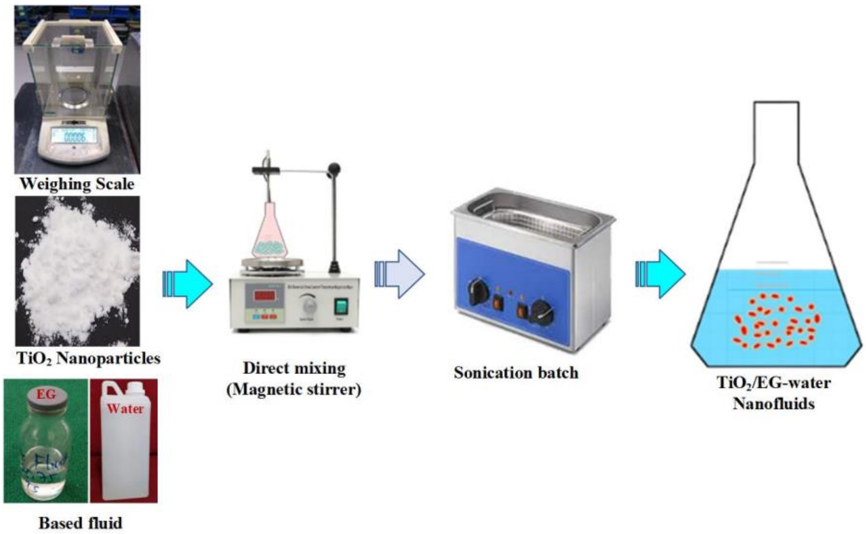

Figure 3. The two-step method approach

X-ray Diffraction (XRD) Examination

XRD data was obtained using a Rigaku maniflex600 with an X-ray wavelength of around 0.15046 nm and a reflection angle adjusted to the characteristics of the TiO_2 particle. The particle structure of rutile TiO_2 has rutile is both thermally and chemically stable [19]. Because of this, it is resistant to changes in structure at high temperatures and aggressive chemical reactions, making it ideal for use in harsh environments. The particle structure of rutile can be displayed and seen in X-ray diffraction (XRD) analysis through the diffraction patterns present in the experiment's findings [20]. The TiO_2 rutile structure encompasses distinct characteristics that can be identified in the position and intensity of the diffraction peaks at approximately 27.4, 36.1, 41.2, and 54.2 in 2theta-degrees [21]. These peaks are available to determine the presence and purity of rutile in samples and to determine particle size and structural properties.

Scanning Electron Microscopy (SEM)

This study employed Scanning Electron Microscopy (SEM) to examine the particle structure of TiO_2 nanoparticles. SEM is a highly efficient technique primarily used to observe objects' particle structure [22] accurately. This technique offers various advantages, such as the ability to achieve nanometer-level high resolution and the simplicity of sample preparation compared to other microscopy techniques like transmission electron microscopy (TEM).

Particle size of TiO_2 Nanoparticle

Diffraction peaks in the XRD data are identified and analyzed using appropriate diffraction analysis software. The diffraction peak width at full-width half maximum (FWHM) of each relevant peak is measured in terms of the corresponding diffraction angle. The Scherrer equation connects the peak width in a diffraction peak (β) to the particle size (D). The larger the particle size (D), the narrower the diffraction peak (β). This phenomenon is used to estimate particle size from experimental diffraction data without having to measure it directly with microscope techniques. Analysis of the particle size of TiO_2 nanoparticles dispersed in EG-water from XRD data was calculated using the Scherrer equation in Eq (1).

$$D = \frac{K\lambda}{\beta.\cos\theta} \quad (1)$$

Where D is the particle size, K is the shape constant whose value ranges from 0.89-9.0, λ is the X-ray wavelength around 0.15046 nm [23], β is the FWHM in radians, and θ is the Bragg angle.

Confirmation test and validations

Spearman correlation is a technique employed to determine the relationship between nanoparticle particle size and other variables without assuming a linear relationship. It is significant because the correlation between particle size and other variables is not always linear. The Spearman correlation coefficient describes and analyzes statistical data, specifically the relationship between FWHM and nanoparticle size.

The Spearman correlation analysis determines the relationship between variables, including size particle and FWHM. Spearman's correlation coefficient (r) ranges between $-1 \leq r \leq +1$ [23]. Positive values indicate that higher levels of one variable are related to higher levels of another. Negative values, on the other hand, suggest that higher levels of one variable are associated with lower levels of another variable. The Spearman correlation formula is calculated using the following equation as Eq (2) [24-25].

$$r = \frac{\sum_{i=1}^{n}\{(x_i\bar{x})(y_i-\bar{y})\}}{\sqrt{\sum_{i=1}^{n}(x_i\bar{x})^2}\sqrt{\sum_{i=1}^{n}(y_i-\bar{y})^2}} \quad (2)$$

Where xi and y_i are the rankings of x and y measurements for each individual of i. Meanwhile, $\bar{x}$ and $\bar{y}$ are the average rankings of measurements x and y, and n is the number of measurement samples.

RESULTS AND DISCUSSION

Diffraction Peak Identification and Peak Width Measurement

The XRD examination yields readily apparent diffraction data with several peaks observed at different Bragg angles (θ) [26]. Each diffraction peak is associated with a specific TiO_2 particle lattice parameter estimation. The FWHM measurement is a crucial indicator in particle size examination, and the results are required to calculate the particle size using the Scherrer Equation [27]. Figure 4 shows that the examination results generated 16 diffraction peaks of TiO_2 nanoparticles.

The particle structure of the rutile of TiO_2 nanoparticle in TiO_2/EG-water can be displayed and seen in the X-ray diffraction results in Figure 3. The diffraction pattern reflects the rutile structure of TiO_2, which is one of the primary focuses of this experiment. The location and intensity of the diffraction peaks in the present study demonstrate the structure of rutile TiO_2, which can be identified at peaks around 27.4, 36.1, 41.2, and 54.2 in 2-degree theta [21]. The diffraction peaks clearly show the typical characteristics of the rutile structure. In this

analysis, distinct diffraction peaks reveal the TiO_2 rutile structure in the sample. A notable diffraction peak appears at an angle of approximately 27.4, 36.1, 41.2, and 54.2 degrees, corresponding to the rutile peaks. The TiO_2 nanoparticle particle structure is consistent with previous research [7, 12].

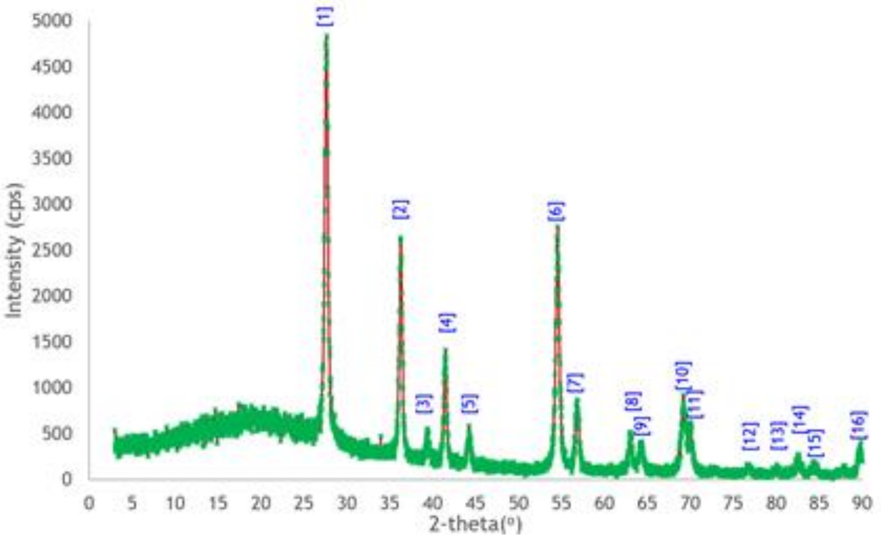

Figure 4. XDR measurement results of TiO_2/EG-water nanofluid

Particle Size Analysis

The Scherrer Equation is used to determine the size of the TiO_2 particle. The TiO_2 particle size in the TiO_2/EG-water sample is calculated using Equation (1). This Equation incorporates the measured FWHM values, the X-ray wavelength (λ) used in the demonstration, and the relevant diffraction peaks' Bragg angle (θ). Figure 5 shows that the examination results show the average particle size of TiO_2 nanoparticles.

The analysis results show that the average particle size of TiO_2 in an EG/water mixture is 19.5 nm, with the largest particle size being 30.38 nm. The standard deviation of this evaluation's results shows the degree of variance in particle size within the sample. The findings of our particle size analysis have significant advantages regarding comprehending the structure of TiO_2 in EG-water mixtures. The average particle size discovered indicates that TiO_2 in this condition has a relatively small particle size compared to the manufacturer's reference and previous research outcomes [12]. These findings outperform previous research that used the reference code JCPDS-211272 and the quadratic particlelization method [11].

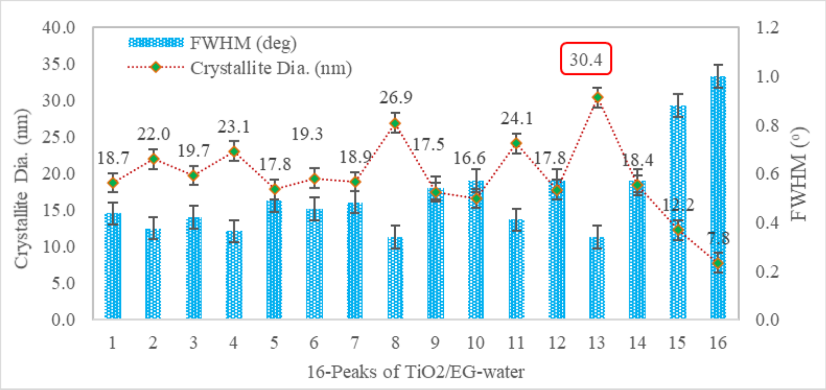

Figure 5. Results of Scherrer Equation analysis of TiO_2/EG-water nanofluid

Scanning Electron Microscopy Analysis

SEM was carried out to see the particle structure of TiO_2 nanoparticles. Anatase and rutile particle line structures are most commonly found in TiO_2 nanoparticles [22]. Figure 6 shows the SEM results of the TiO_2 nanoparticles' rutile particle structure. Rutile particles in nanoparticles have a tetragonal particle structure [19].

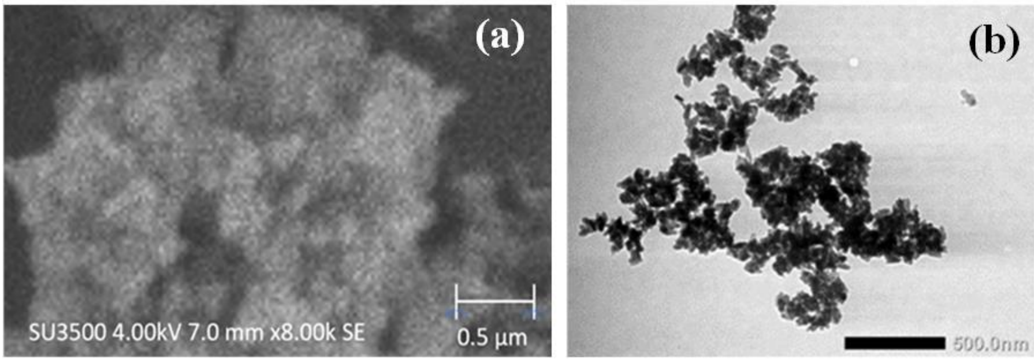

Figure 6. SEM image of TiO_2/EG-water nanofluid: (a) Magnification 8k (b) Magnification 10k

Statistical Data Validation

An analysis of diffraction was performed on the TiO_2 nanoparticles dispersed in EG-water, as stated in previous sections. This study revealed the presence of sixteen identifiable diffraction peaks in the TiO_2 sample. Each peak's particle size (D) was determined using the Scherrer equation. Table 1 presents the data acquired by measuring the particle's size, as depicted in Figure 5, along with the probability plot illustrated in Figure 7.

The subsequent step involves statistical data analysis using a probability plot approach and variable correlation analysis. The input data in correlation analysis has been adjusted to achieve a confidence level of 95% using statistical software. It is essential to ensure the accuracy and reliability of the generated data. Statistical data analysis entails computing the mean value of particle size and its corresponding standard deviation. The standard deviation quantifies how much these values deviate from the mean. The statistical data analysis was conducted using specialized software, with a confidence level established at 95%. The results of statistical data analysis are presented in Figure 7. Statistical analysis shows that the average particle size of TiO_2 nanoparticles is about 19.45 nm, with a standard deviation of about 5.32 nm.

Table 1. The Scherrer equation results from the particle size of TiO_2 nanoparticles

Peak No.	2-theta(deg)	Particle Dia. (nm)
1	27.60	18.72
2	36.24	21.97
3	39.37	19.75
4	41.42	23.07
5	44.21	17.81
6	54.49	19.34
7	56.77	18.87
8	62.95	26.94
9	64.19	17.45
10	69.16	16.61
11	69.97	24.12
12	76.63	17.78
13	80.05	30.38
14	82.62	18.39
15	84.53	12.25
16	87.90	7.76

The results show a deviation of the particle size of about 5.32 nm in the TiO_2 nanoparticle sample relative to the average. The data shows an inconspicuous dispersion in TiO_2 particle dimensions, with specific nanoparticles displaying relatively close sizes while others are close to the average. This data provides more accurate results than research using the reference code JCPDS-211272 and the quadratic particlelization system reported by [11]. The study indicates that TiO_2 employed a particle size of approximately 30 nm, while the analysis findings obtained through this method vary between 33.47 - 73.65 $\dot{A}$˙, equivalent to approximately 3.35-7.37 nm.

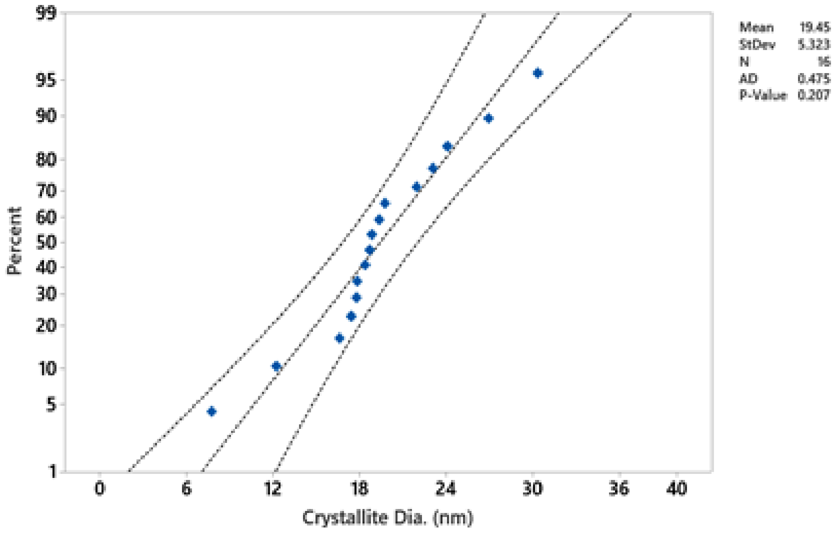

Figure 7. Probability plot of size particle line TiO_2/EG-water

Statistical software was utilized to conduct correlation analysis, unveiling the factors impacting nanoparticle particle size. Subsequently, a Spearman correlation coefficient of 0.958 was derived. The correlation between nanoparticle particle size and their full width at half maximum (FWHM) was inversely proportional. Figure 8 illustrates the outcomes of the Spearman correlation analysis.

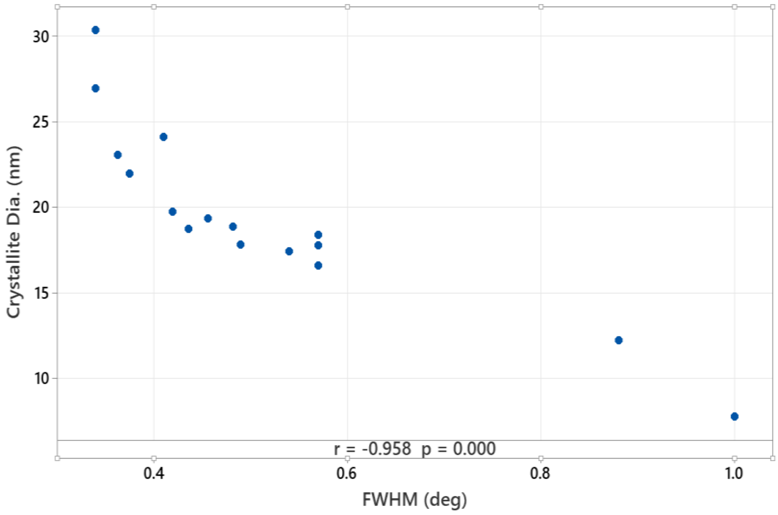

Figure 8. The Spearman correlation FWHM versus Particle size of TiO2 nanoparticle.

CONCLUSION

The study successfully measured the size of TiO_2 particles dispersed in a mixture of EG and distilled water using XRD data and the Scherrer equation. The average particle size observed in the samples is approximately 19.45 nm, with a deviation of about 5.32 nm. These findings affirm that the Scherrer equation, coupled with XRD data, offers a viable method for analyzing nanomaterial sizes. Moreover, this method validates that the nanomaterial fulfills the requirements as nanoparticles, given that the particle size of TiO_2 nanoparticles in TiO_2/EG-water remains below 100 nm, indicating relatively small particle dimensions compared to specified standards. Analysis of TiO_2 particle size using the Scherrer equation reveals a negative Pearson correlation level of -0.958, indicating that higher FWHM values correspond to smaller particle sizes. Determining particle size using the Scherrer equation with XRD data has greater accuracy than other methodologies, such as JCPDS-211272 and quadratic particlelization. These results and correlations offer valuable insights into the particle structure of TiO_2 and its implications for TiO_2-based nanomaterial development, as well as broader applications in nanomaterial characterization. Future research will explore the more general applications of TiO_2 in diverse technological fields.

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