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The Effect Of Composition Variation On The Functional Groups Of Coconut Husk Ash Silica Nanocomposites With Titanium Dioxide

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Abstract: Studie has been performed with the title Effect of Composition Versions on Useful Corporations of Coconut Coir Ash Silica Nanocomposites with Titanium Dioxide. This study goals to determine the traits of silica gel from the processing of coconut husk ash modification TiO_2 in phrases of useful groups variations in silica composition with titanium dioxide, particularly 3.5: 1.5 and 4: 1 (grams). The synthesis of silica gel from coconut husk ash become finished the use of the gel sol method. FTIR (Fourier Transform Infrared) characterization effects show the achievement of TiO_2 changed silica gel and silica gel synthesis proven with the aid of the absorption of Si-O-Si and Si-OH in silica gel and Ti-O-Ti absorption in TiO_2 changed silica gel. Based on the FTIR spectrum, it was obtained that during all samples an ordinary practical group of TiO_2 seemed, specifically Ti-O at an absorption peak of about 500-600 cm^{-1} O-H vibrational bond stretching and O-H bending at an absorption height of 1500 - 3500 cm^{-1}. Appeared in all samples indicating the presence of water molecules. In addition, the occurrence of vibrational bond between O-C-O and C-H indicating the use of organic materials NaOH and HCL in the extraction process. After the composite process was carried out, the C=C functional group appeared in the TiO_2 and SiO_2 composites at variations of 3.5 : 1.5 and 4 : 1 (grams) respectively, indicating the presence of carbon bonds originating from coconut coir ash.

Keywords: Coconut Coir Ash; TiO_2; Functional Groups; Nanocomposite; Silica Gel.

1. Introduction

Composite materials have not developed so rapidly and have not been widely applied in Indonesia. However, this material is actually very efficient to use because if the composite material is formed, the physical and chemical properties of this composite will be better than the constituent material [1]. These unique properties can be altered by controlling material size, chemical composition, surface morphological modifications, and interactions between particles by merging two or more materials. Titanium dioxide, additionally known as titanium (IV) oxide or titania, is an oxide of titanium, with the molecular formula TiO_2. Has a molecular weight of 79.90 g/mol; Density 4.26 g/cm-3 [2]. TiO_2 does now not soak up visible mild however is able to soak up UV radiation so that it is able to purpose hydroxyl radicals in pigments as photocatalysts [3]. Photocatalyst mechanism based totally on the absorption of light (spectrum) with the aid of semiconductor materials in order that electron pairs and holes are shaped on the surface [4]. Photocatalysts are often used with TiO_2, due to the fact it is cheaper and environmentally pleasant [5].

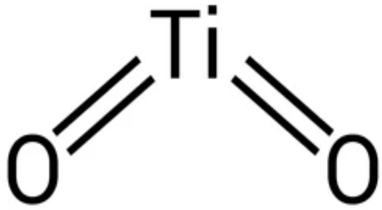

Figure 1. Titanium Dioxide

The catalytic activity of the catalytic interest of titanium oxide is stimulated by means of crystal shape, surface place, particle size distribution, porosity and surface Tanium oxide is prompted by means of crystal structure, floor location, particle size distribution, porosity and surface. Pure TiO_2, with a metastable anatase crystal structure having a wide gab band (3.2 eV) has made it a predominantly UV-activated photocatalyst due to its stability and high oxidation potential and chemically beneficial properties [6]. Various ways can be done to minimize the energy gap, such as heating treatment by calcination, modification of material composition and doping of material with other materials. One way to reduce the energy gap is to do other materials to TiO_2, material. For doping can use transition metallic substances together with Fe, Au, Mo, and others[7][8][9]. It could additionally be non-metallic materials which include C, N, and S [10], [11][12].

The photocatalytic activity (photoactivity) of TiO_2 can be increased through the development of supporting materials [13]. The combination of TiO_2, with one of the supporting materials will form a composite. a few helping substances that are frequently used are silica [4]. This supporting material serves to reduce the energy gap in TiO_2, so that it is easier to activate. In addition to reducing the energy gap, the addition of silica can also increase thermal stability [14].

Coconut coir can be used as uncooked fabric for producing ash through the smelting technique, as lots as 33%-35% of coconut coir capabilities as a mesocarp of coconut fruit [15]. Coconut husk ash includes 42.98% silica: 2.26% aluminum and 1.16% iron [16]. Coconut husk ash also can be used for the manufacture of silica gel in which in this silica gel there may be an lively group of silanol (≡Si-OH) and siloxane (≡Si-O-Si≡) [17]. Silica gel is one of the silica-based substances that has extensive uses including within the pharmaceutical enterprise, ceramics, paints, and unique applications in the chemical area. Silica gel is a polymer of silicic acid with a large molecular weight and absorbs a whole lot of water so that it's far a chewy stable. Another definition of gel silica is amorphous silica consisting of irregular tetrahedral SiO_4 globules that aggregate to form a three-dimensional skeleton [18][19].

Figure 2.Coconut Coir

Silica gel is an inorganic solid that has an energetic facet Si-O-Si practical organization and a Si-OH [20]. Practical organization the lifestyles of these practical businesses makes silica gel potentially used as a [21]. adsorbent the potential to soak up silica gel adsorbents is stimulated by using the Si-OH institution and the Si-O-Si group [22]. In trendy, silica gel is used as a desiccant in food and non-food products. Silica gel is inert, non-toxic, non-flammable, does no longer reason chemical reactions whilst soaking moisture, and can be used again and again, making it greater economically fine [23].

The use of silica as a cellulose silica composite fiber has also experienced rapid development, where the composite fiber is widely used in industrial fields such as the production of medicines, catalysts, ceramics, pigments, biotechnology, photonics, biomedicine and various other technical fields. Cellulose silica fiber can be made as a raw material to resist structural deformation and thermal insulation materials, with a higher modulus of elasticity and tensile strength. The surface of silica cellulose fibers can also be used to retain water very well [24].

The success of the bonding between TiO_2 and SiO_2 can be determined by examining the functional groups present in the sample. These functional groups involve interactions between the molecular bonds of TiO_2 and SiO_2. Analysis on these functional groups can be performed by characterizing the sample using an FTIR (Fourier Transform Infrared) spectrometer. When infrared electromagnetic waves hit a material, an interaction occurs in the form of absorption of energy by the atoms or molecules of that material, causing vibrations of the atoms in those molecules. TiO_2 and SiO_2 have potential applications as photocatalysts, with TiO_2's energy gap decreasing after variation. However, previous research has not given brief discussion on functional groups. Therefore, a discussion regarding the effect of varying compositions of TiO_2 and SiO_2, with ratios of 3.5:1.5 and 4:1 (grams), is conducted. Subsequently, to determine the bonding between TiO_2 and SiO_2 in the sample, tests are carried out using FTIR spectroscopy.

2. Materials and Method

This research consists of several levels beginning with coconut husk ash, coconut husk ash silica extraction, coconut husk ash silica composite education with TiO_2, characterization and assessments finished to decide the practical corporations and wavenumbers of coconut husk silica nanocomposites with TiO_2,. The equipment used on this have a look at are *Fourier transform infrared* (FTIR), oven, stirrer. The ingredients used in this examine were coconut husk ash, TiO_2, 0.1 M sodium hydroxide solution, 0.1N hydrochloric acid solution and aquades Figure 2.

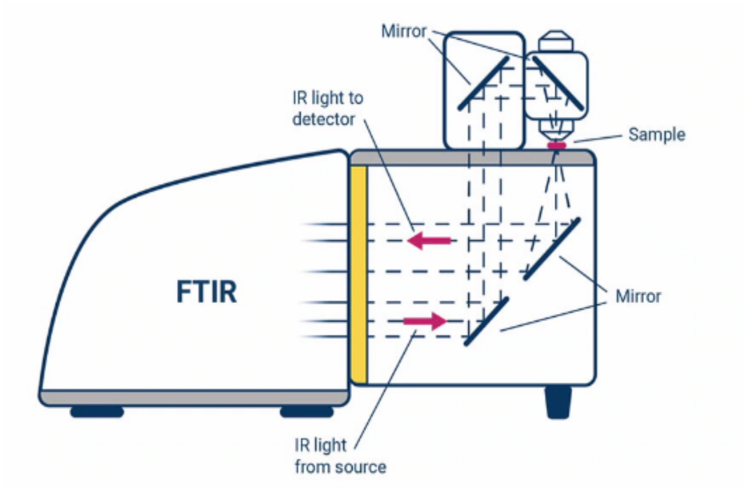

Figure 3. How FTIR (Fourier Transform Infrared)

2.1. Greying of Coconut Coir Ash

Coconut husk is washed repeatedly until clean and then is heated in an oven at 120 $°C$. Then the coconut husk ash is furnaced with a temperature of 600 $°C$ for 2 hours. Then the coconut husk ash that has been furnaced is mashed using *milling* for 3 hours until it forms nanoparticle grains as shown in Figure 4.

Figure 4. Coconut husk ash after milling

2.2. Silica Extraction from Coconut Coir Ash

Coconut husk ash weighed as much as 5 grams put into a beaker and added 0.1M NaOH as much as 100 ml. Then stirred using a stirrer at a temperature of 700 $°C$ for 40 minutes. Furthermore, the coir ash is filtered using filter paper to separate the coconut husk residue from sodium silicate. The resulting sodium silicate is delivered 30 ml of HCL 1N stirred using a magnetic stirrer at a temperature of 60 $°C$ for 40 minutes, then prompted for 24 hours with aquades. The result of the precipitate is repeated washing with aquades until a neutral Ph is received. the sediment is then dried the use of an oven with a temperature of 105°C for 2 hours.

2.3. Preparation of Coconut Husk Ash Silica Composite with TiO_2

Where every 2.5 gr and 3 gr; brought to a beaker as tons as 50 ml NaOH 0.1 M, mixing using a magnetic stirrer with a stirring pace of 600 rpm for 50 minutes. TiO_2 The dissolved ones are blended with silica every 2.5 gr and 2 gr stirred at a speed of 600 rpm with a temperature of50$°C$ for 50 minutes. Then the composite answer TiO_2-SiO_2 within the oven to obtain a composite powder. Composite powder TiO_2-SiO_2 the resulting one is characterized through FTIR (*Fourier Transform Infrared)* to achieve its wavenumber and functional businesses.

Functionality group characteristics are used to identify mixtures of chemical bonding compounds as well as components of the organic content of a material that can be analyzed using FTIR (*Fourier Transform Infrared).* Characterization using FTIR Spectrometer tool with infrared spectrum middle region wavenumber range 400-4000 cm^{-1}. The characterization results have been analyzed for every purposeful organization by way of comparing the results in opposition to the wavenumber table and functional companies in the FTIR (*Fourier Transform Infrared)* spectrum from the results of previous studies. The results of the analysis are graphs of the relationship between wavelength and transmittance.

Based totally on the general silica cellulose merchandise produced, only a few were usually examined using IR Spectrophotometry (FTIR) to decide organic and inorganic purposeful groups, and the excellent situations were received at pH = 7. Because at pH = 7 gel formation is highly faster than gel formation at pH 7. The maximum fee of polymerization takes place at pH = 7 (impartial) due to its minimum stability. Balance right here approach the opportunity of precipitation of silica acid answer via saturation [25]. Below various modulus conditions with a uniform pH (pH = 7), it is able to be found that there are numerous functional groups of the five silica cellulose sample merchandise having similar spectra. The consequences of the spectra analysis confirmed that there have been several practical group compounds recognized along with alcohol purposeful corporations (-OH), carboxyl practical businesses (-COOH), siloxane useful businesses (Si-O-Si) and Silanol practical organizations (Si-OH).

3. Results and Discussion

3.1. Useful businesses and wavenumbers of coconut husk ash silica nanocomposites with TiO_2

That is additionally bolstered with the aid of Studie that has been conducted by using [26] that the quantity of TiO_2 included in the silica matrix increases with the addition of increasingly titania. Composite characterization consequences TiO_2-SiO_2 with variations of 3.5 : 1.5 / g range 400 - 4000 cm^{-1}, Figure 3 suggests the FT-IR spectral purposeful group in say wave 3328.32 cm^{-1} and the transmittance of 35.54% is the OH extended vibration of Ti-OH / Si-OH. The O-H bend vibrational institution of Ti-OH/Si-OH takes place in say wave 1637.85cm^{-1} and transmittance 40.20%, vibrational functional group Si-O-Si bond at wavenumber 1058.30cm^{-1} and transmittance 32.26%, vibrational functional group Ti-O-Ti bond at wavenumber 529.25cm^{-1} with a transmittance of 30.34% [27].

Table 1. Wavenumber and transmittance of silica nanocomposites TiO_2-SiO_2 3,5 : 1,5/grams)

Peak number	**Wavenumber (cm^{-1})**	**Transmittance(%)**
1	3336.08	39.16
2	2336.27	46.89
3	1638.02	43.05
4	1396.67	45.21
5	1063.96	35.69
6	529.25	30.34
7	455.15	49.04

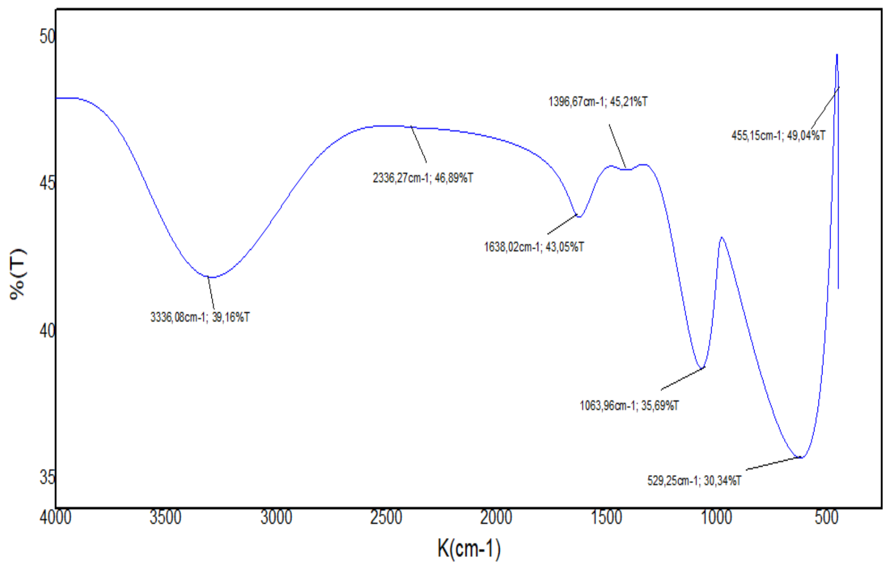

Figure 5. Nanocomposite FTIR Data Analysis TiO_2-SiO_2(3.5:1.5/g)

Composite FT-IR spectrum transmission TiO_2-SiO_2 with variations of 3.5 : 1.5 / g range 400 - 4000 cm^{-1} , Figure 4 shows the FT-IR spectral useful organization in say wave 3262.88 cm^{-1} and the transmittance of 41.57% is the OH extended vibration of Ti-OH / Si-OH. The O-H prolonged vibrational functional group of Ti-OH/Si-OH occurs in say wave 1637.30 cm^{-1} and transmittance 47.33%, vibrational functional group Si-O-Si bond at wavenumber 1068.56 $cm^{-1}$and transmittance 41.26%, vibrational functional group Ti-O-Ti bond at wavenumber 536.035 cm^{-1} with a transmittance of 34.63%.

Table 2. Wavenumber and Transmittance of Silica Nanocomposites TiO_2-SiO_2 (4 :1/grams)

Peak number	**Wavenumber (cm^{-1})**	**Transmittance (%)**
1	3262.88	41.57
2	2342.86	51.16
3	2108.5	51.71
4	1637.3	47.33
5	1390.92	49.69
6	1068.56	41.26
7	536.03	34.63
8	488.7	53.85
9	435.57	54.26

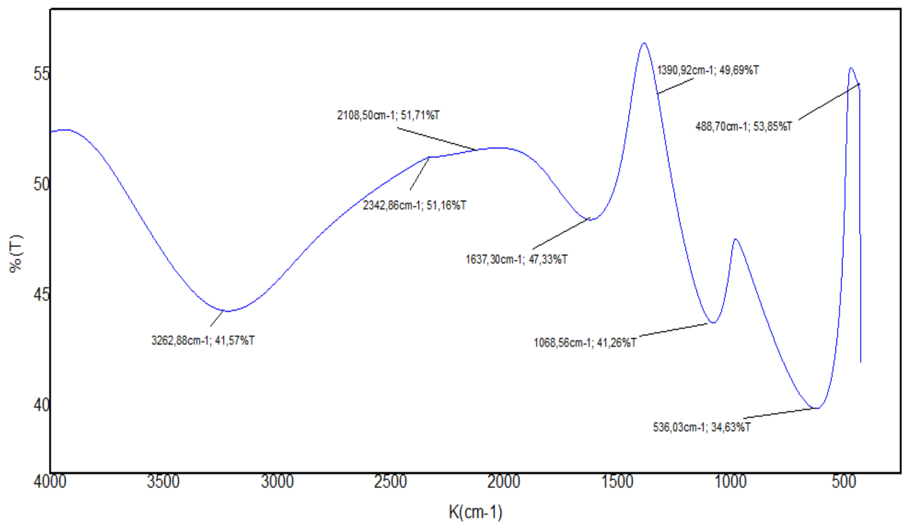

Figure 6. Nanocomposite FTIR Data Analysis TiO_2-SiO_2 (4:1/g)

3.2. *Influence of composition variations TiO_2-SiO_2 on wavenumbers and functional groups*

The IR spectroscopy results in Table 1 show a change in the absorption band (wavenumber) of the composite TiO_2-SiO_2 4 : 1 (grams) variation which is 3262.88 cm^{-1} becomes wider on composites cm^{-1} TiO_2-SiO_2 variation of 3.5 : 1.5 (grams) which is

3328.32 cm^{-1}. This widening indicates the vibration of the OH group from silica, a decrease in absorption intensity at wavenumbers 1500-1300 cm^{-1} nanocomposites show the interaction between Ti and Si, namely in the form of hydrogen bonds between the OH group and the silanol group (Si-OH) with the Ti-OH group. The results of analysis by infrared spectroscopy only provide information about the absorption of functional groups so that in general the spectra of Origin Silica and TiO_2-Silica are almost the same. The absorption characteristics of the functional group of TiO_2-SiO_2 can be seen in Table 3.

Table 3. Functional Groups of Silica Nanocomposite Variations TiO_2- SiO_2

Functional Groups	Wavenumber (cm^{-1})			
	$3,1 : 1,5\ (grams)$		$4 : 1\ (grams)$	
	TiO_2	SiO_2	TiO_2	SiO_2
Extended vibration – OH from Ti-OH or Si-OH	3262,88	3262,88	3262,88	3262,88
Bending vibration-OH of Ti-OH or Si-OH	1637,85	1637,85	1637,30	1637,30
Asymmetric extended vibration Si-O-Si	-	1058,30	-	1068,56
Asymmetrical extended vibration Ti-O-Ti	529,25	-	536,035	-

The maximum transmittance relationship on 2 compositional variations of the nanocomposite - can be seen in Figure 5. Where the average transmittance frequency is 50%. This indicates there are some IR frequencies absorbed by the compound and there are 50% IR frequencies passed. The higher the uptake can provide information about the dominant functional group.

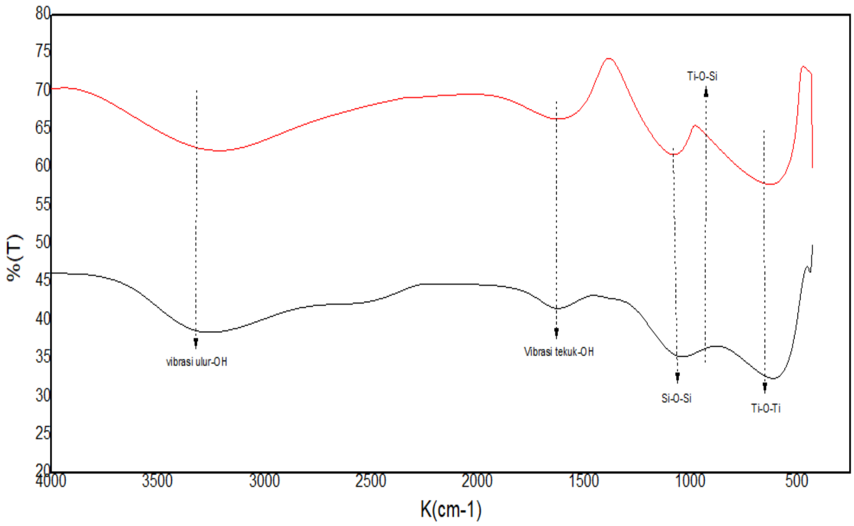

Figure 7. Nanocomposite FTIR Data Analysis TiO_2-SiO_2

The FTIR spectra of the whole nanocomposite-coated fabric in Figure 5 showed no significant change compared to the variation in composition. This is due to overlapping peaks among the layers and the width of the cellulose band of the cloth. A covalent bond is shaped between the give up of the silica titanium organization and the hydroxyl institution on the cellulose fiber, therefore making the silica titanium layer more durable. The results display that the adhesion of the titanium silica nanocomposite layer to the surface of the fabric is in principle inspired by way of the chemical composition and structure of the material substrate [28].

The difference between the two FTIR spectra is in the peak absorption shift as presented in table 3. The difference between the two spectra is in the addition of TiO_2 mass. The presence of TiO_2 in silica affects the shift of absorption peaks and decreases the intensity of absorption peaks as the mass of TiO_2 is added as in the decrease in absorption intensity at the bending vibrational peaks -OH at wavenumbers 1637.85 cm^{-1} and 1637.30 cm^{-1}. This is due to the presence of steric obstacles that arise as the mass of TiO_2 increases and inhibits the mobility of the charge carrier, causing the conjugation chain to get shorter [29].

The anticorporation between titanically composites not only interacts physically (titania not only attaches to silica shells) but also interacts chemically with the formation of SiO-Ti groups in the results of FTIR.

The presence of a peak at wavenumber 3262.88 cm^{-1} indicates the presence of free Si-OH and -OH groups. The wave crest at 1637.85 cm^{-1} shows wave absorption from the C=C group which can also be attributed to the aromatic group possessed by citric acid which is difficult to separate during the washing process. The Si-O-Si silica functional organization is proven by wave vibrations of 1058.30 cm^{-1} to 1068.56 $cm^{-1}$and at waves of 890 cm^{-1}a Ti-O-Si practical group is acquired that is feature of titania-silica composites. It has been established via FTIR evaluation on both variations acquired by purposeful organizations with distinct intensities, and additionally received in waves 529.25 cm^{-1} and 536.035 cm^{-1}, specifically within the shape of TiO_2 groups, primarily based on discern, it may be concluded that the form of variation used will substantially have an effect on the organization fashioned. Because, from each element forming the composition of the composition given will be bound by TiO_2 and then composite by Na_2SiO_3 will be able to form salt elements or other elements even though it has gone through the process or washing stage. The clusters captured at the intensity of these waves show that silica has bonded to titania and formed almost similar properties and characters in some variation in composition. So from the consequences of this FTIR it's far concluded that versions in composition of 3.5: 1.5 (grams) and 4: 1 (grams) show the effects of wave emission that are not tons one of a kind, most effective specific in the stretch because of the composition of tio2 in its capability to bind sodium businesses to Na_2SiO_3 which ends up in stress on the silanol group (Si-OH).

The results of nanocomposite data / on the FTIR characterization tool show the relationship between the wavenumber (cm^{-1}) and %Transmittance. Based on FTIR data analysis, peaks were obtained which showed that silica has an active OH group bound to silanol which is at wavenumber 3262.88 cm^{-1}. Silica has active groups on its surface, namely the silanol group (Si-OH) and the siloxane group (Si-O-Si). The O-H group bound to silanol is replaced by the appearance of an absorption band which is the Si-O extended vibration of Si-OH at 1058.30 cm^{-1}which indicates that silica as a coupling agent has created a crosslink. So that in wave 536.035 cm^{-1}obtained the functional group Ti-O-Si which is characteristic of composite. From the results of FTIR spectra it can be concluded that silica has been distributed into TiO_2 and has succeeded in becoming a link in O_2 : SiO_2 nanocomposites so that it can add mechanical properties to TiO_2.

Figure 5 shows the functional groups of the FT-IR spectrum in say bands 3500-3000 cm^{-1} is the outstretched vibration OH of Ti-OH /Si-OH. The O-H bend vibrational functional group of Ti-OH/Si-OH occurs in say waves 1500-1300 cm^{-1} , Ti-O-Si bond vibrational functional groups at wavenumbers 1000-900 cm^{-1} which indicates a chemical reaction between TiO_2 and SiO_2 forms Ti-O-Si bonds. Ti-O-Si bonds appear in all samples signaling successful composite synthesis.

IR spectroscopy results showed a change in the composite absorption band TiO_2 - SiO_2 4:1 (grams) variation about 3264 cm^{-1} becomes wider on composites TiO_2 - SiO_2 variation 3.5 : 1.5 (grams) about 3361 cm^{-1} This widening indicates the presence of an extended vibration of the OH group from silica. The occurrence of a decrease in absorption intensity at wavenumbers 1500-1300 cm^{-1} nanocomposites show the interaction between Ti and Si, namely in the form of hydrogen bonds between the OH group and the silanol group (Si-OH) with the Ti-OH group.

The FTIR spectra of the whole nanocomposite-coated fabric showed no significant change compared to compositional variations TiO_2 - SiO_2 . This is due to overlapping peaks between the layers and the width of the cellulose band of the fabric. A covalent bond is formed between the end of the silica titanium group and the hydroxyl group on the cellulose fiber, thus making the silica titanium layer more durable. The results show that the adhesion of the titanium silica nanocomposite layer to the surface of the fabric is in principle influenced by the chemical composition and structure of the fabric substrate.

The analysis results from figure 51 show that there is absorption at wavenumbers 3300-2200 cm^{-1} which is the elongation vibration –OH of the SiOH and TiOH groups. The wavenumbers generated in this absorption region vary each nanocomposite TiO_2 - SiO_2 i.e. an average of 3300cm^{-1} . Average wavenumber 500 cm^{-1} of each nanocomposite TiO_2 - SiO_2 is a Ti-O-Ti bend vibration. As for each Si-O-Si bend vibration of the nanocomposite TiO_2 - SiO_2 i.e. an average of 450 cm^{-1}. Based on the data analysis, it can be said that the synthesis of nanocomposites TiO_2 and SiO_2 is succeed.

In all variations of nanocomposites it was seen that on average half the transmittance frequency was absorbed. This indicates there are multiple IR frequencies absorbed by the compound and there are IR frequencies being passed. The higher the uptake can provide information about the dominant functional group.

Self-cleaning activity depends on the peaks of the Ti-O-Si bond formed. The FTIR results are reinforced by the results of the diffractogram at the peak of 2θ which shows that the addition of SiO_2 to the composite can reduce the crystallinity properties of TiO_2 - SiO_2, so it can be concluded that composites with different variations have no significant effect.

4. Conclusion

Functional group analysis of TiO_2 - SiO_2 samples with nanocomposite composition of 3.5:1.5 and 4:1(grams) versions showed nearly the equal FTIR spectral sample. inside the complete sample, a normal purposeful institution of TiO_2 seems, namely Ti-O at an absorption peak of round 500-600 cm^{-1}. In addition, O-H stretching and O-H bending vibrational bonds also seem which indicate the presence of water molecules in the sample. Then there is a Si-O-Si vibrational bond which indicates the addition of silica to the synthesis process. The presence of silica is shown in the Ti-Si vibrational bond that appears in the sample of all samples after being composite with TiO_2 indicates the success of silica composites with TiO_2

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