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**Preparation of Titanium Oxide, Iron Oxide, and Aluminium Oxide
from Sludge generated from Ti-salt, Fe-salt and Al-salt Flocculation of
Wastewater**

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Abbreviated title: Preparation of metal oxides from sludge

Abstract

In this study, the settled floc (sludge) produced by aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$), ferric chloride (FeCl_3) and titanium tetrachloride (TiCl_4) flocculation was recycled with a novel flocculation process, which has a significant potential to the lower cost of waste disposal, protect the environment and public health and yield economically useful by-products. Three coagulants removed 70% of organic matter in synthetic wastewater. The settled floc was incinerated in the range from 100 °C to 1000 °C. Alumina (Al_2O_3), hematite (Fe_2O_3), titanium oxide (TiO_2) which are the most widely used metal oxides were produced from the wastewater sludge generated by the flocculation in wastewater with $\text{Al}_2(\text{SO}_4)_3$, FeCl_3 and TiCl_4 , respectively. TiO_2 particles produced from the sludge

consisted of the large amount of nano size particles. Hematite (Fe_2O_3) and grattarolaite ($\text{Fe}_3(\text{PO}_4)_2$ or Fe_3PO_7) included the majority of micro size (40%) particles. Alumina (Al_2O_3) also consisted of micro size (40%). Due to TiO_2 usefulness of the application, detailed characterisation of TiO_2 after calcination at different temperatures were investigated in terms of X-ray diffraction, energy dispersive X-ray, surface area and photoactivity.

Keywords: Flocculation, sludge recycling, titanium dioxide, hematite, alumina

1. Introduction

Flocculation causes contaminants to coagulate and is one of the most common chemical treatment methods. Flocculation can be used to remove organic matter, which causes trihalomethane formation during disinfection. However, the flocculation process using a coagulant produces a large amount of sludge which reduces the efficiency of wastewater treatment [1]. Most of this sludge is solid waste from which nothing beneficial can be recovered, cannot be reused and requires further treatment such as incineration, landfill, etc. A novel coagulant and/or a novel process that produce less sludge or more reusable sludge offers a solution to many environmental and economic problems associated with sludge handling.

Commonly-used coagulants include aluminum (72%) and iron salts (23%) in water treatment plants [2]. Recently, Shon et al. [3] developed a new coagulant which is titanium tetrachloride (TiCl_4). The alternative coagulant (TiCl_4) changes into TiOCl_2 in

wastewater. Depending on pH, the TiOCl_2 hydrolyses to Ti(OH)_4 . As the zeta potential of the negatively-charged organic matter is broken by Ti(OH)_4 , organic matter aggregates with the Ti(OH)_4 . Here phosphorus and a variety of metal compounds are found in the wastewater. During incineration of the settled floc, 55% of water and 20% of organic matter in the wastewater are vaporized and resulting in the compound TiO_2 . Titanium chloride (TiCl_4) has widely been used in industry as an intermediate in the production of titanium rutile, titanium oxide and titanium pigments. Importantly, the cost of bulk TiCl_4 is comparable to iron chloride or aluminium sulphate so the process offers an efficient and economical alternative for the removal of organic matter and sludge reduction. Furthermore, protocols for the safe handling and use of bulk TiCl_4 are well documented [4]. Thus, the TiCl_4 flocculation is efficient and economical not only in terms of removal of organic matter, but also reducing the amount of sludge.

The objective of this study is to reduce the sludge produced from a flocculation process and produce useful byproducts by calcination of sludge. Byproducts produced from sludge obtained after Fe, Al and Ti-salt flocculation were investigated in term of particle structure, functional group, elemental content and particle size.

2. Experimental

2.1 Synthetic wastewater and organic removal by flocculation

Flocculation was conducted with different coagulants (TiCl_4 , FeCl_3 , and $\text{Al}_2(\text{SO}_4)_3$) with an optimum dose in synthetic wastewater. The composition of the synthetic wastewater is presented in Table 1 [5]. The relative molecular mass of the mixed synthetic wastewater ranged from 300 daltons to about 34100 with the highest fraction of 900 – 1200 daltons. Although sodium lignin sulphonate and tannic acid showed the peaks at 12100 and 6300 daltons respectively, the corresponding peaks were not found in the mixed synthetic wastewater. It may be due to aggregations between organic and inorganic compounds in the synthetic wastewater. The samples were stirred rapidly for 1 minute at 100 rpm, followed by 20 minutes of slow mixing at 30 rpm, and 30 minutes of settling. Organic matter was measured using a Dohrmann Phoenix 8000 UV-persulphate TOC analyzer equipped with an autosampler. All samples were filtered through 0.45 μm membrane prior to organic measurement.

Table 1. Constituents of the synthetic wastewater

2.2 Characterisation of the settled floc (sludge) and byproduct (metal oxide)

The decantability test with different settled flocs after flocculation was measured using the ratio of floc head in terms of length unit (cm). XRD images (Rigaku, Japan) of anatase and rutile TiO_2 photocatalysts to identify the particle structure was investigated. All the XRD patterns were analyzed with MDI Jade 5.0 (Materials Data Inc., USA). UV-VIS-NIR spectrophotometer (Cary 500 Scan, Varian, USA) was used to identify the absorbance range and the band gap of TiO_2 incinerated at different temperatures was calculated. The photocatalytic activity test of TiO_2 was investigated under irradiation of

UV (Sankyo, F10T8BLB, three 10 W lamps) and visible light (Kumbo, FL10D, three 10W lamps) using the method of photodecomposition of gaseous acetaldehyde. The concentration of acetaldehyde was measured by gas chromatography (Youngin, M600D, Korea). The particle size distribution of metal oxides after calcinations was investigated with a laser particle size analyzer (LS-230, Beckman Coulter, USA). The visual microscopy was used to measure the shape and size of TiO₂. SEM/EDX results were investigated. Micromeritics Gemini 2360 analyzer (USA) for BET surface area analysis was used with automatic surface area analyzer.

3. Results and Discussion

3.1 Organic removal by Ti, Fe and Al-salt flocculation and decantability test of the settled floc

Fe, Al and Ti-salt flocculation at optimum concentration using synthetic wastewater was investigated in terms of organic removal (Figure 1). Removals of organic matter by Fe, Al and Ti-salt flocculation were found to be 73% (13.8 Fe-mg/L), 70% (16 Al-mg/L) and 70% (9.8 Ti-mg/L), respectively. Three coagulants had the range of similar organic removal. Figure 2 shows the decantability test with different flocs. Here, the ratio can be defined as floc head in terms of length unit (cm). The Fe floc settled fast down compared with Ti and Al flocs. The decantability decreased in the order of Fe floc > Ti > Al.

Figure 1. Organic removal by different flocculations with synthetic wastewater (initial organic conc. = 11.5 mg/L)

Figure 2. Decantability ratio of different flocs

3.2 XRD patterns of metal oxides obtained after calcination of the settled floc

Figure 1 shows XRD images to identify the particle crystalline structure after calcination of the settled floc at different temperatures. After TiCl_4 flocculation, the anatase pattern was found from more than 600 °C (Figure 1a). At lower temperature, remaining organic matter interfered TiO_2 production. At 1000 °C, the anatase structure changed to rutile. Although there are a lot of compounds in synthetic wastewater, genuine TiO_2 crystalline structure was found. In the case of the settled floc after FeCl_3 flocculation, the incinerated flocs were found to have different structures (hematite (Fe_2O_3) and grattarolaite ($\text{Fe}_3(\text{PO}_4)\text{O}_3$ or Fe_3PO_7) (Figure 1b). On the other hand, only Al_2O_3 was made at 1000 °C after $\text{Al}_2(\text{SO}_4)_3$ flocculation (Figure 1c). Interestingly, berlinite (AlPO_4) could not be made with Al- flocs. These metal oxide by-products produced in such a way are efficient and economical not only in terms of removal of organic matter in wastewater, but also in sludge reduction. As the metal oxides can be produced in significant quantities in wastewater treatment plants, they can also easily meet demands for different metal oxides in other applications such as environmental photocatalysis.

Figure 3. XRD images a) of the settled floc after TiCl_4 flocculation, b) of the settled floc after FeCl_3 flocculation (hematite (Fe_2O_3): 24° , 33° , 35° , 40.5° , 49.4° , 54° , 57.5° , 62.5° , 63.9° , 71.8° , 75.3° ; grattarolaite ($\text{Fe}_3(\text{PO}_4)\text{O}_3$ or Fe_3PO_7): 18.2° , 28.9° , 36.8° , 38.9° , 43.3° , 45.3° , 45.6° , 49.2° , 55.1° , 56.7° , 59.7° , 60.1° , 61.5° , 64.7° , 70.4°) and the settled floc after $\text{Al}_2(\text{SO}_4)_3$ flocculation (Al_2O_3 : 32.3° , 34.6° , 36.9° , 39.0° , 45.0° , 46.3° , 60.8° , 67.8°) by calcination at different temperatures

3.3 Particle size distribution of metal oxides

Figure 4 shows particle size distribution with different metal oxides produced from sludge. The majority of particles included a broad range of size distribution. TiO_2 calcinated at 600°C produced a high proportion of nm size particles (10 – 25 nm) of up to 25%. Hematite (Fe_2O_3) and grattarolaite ($\text{Fe}_3(\text{PO}_4)\text{O}_3$ or Fe_3PO_7) produced by Fe-salt flocculation (700°C calcination) consisted of nano size particles (5%) and 2.75 μm micro size particles (40%). Alumina (Al_2O_3) from Al-salt floc (1000°C calcination) consisted of nano size particles (5%) and 2.75 μm micro size particles (40%). Since TiO_2 is an attractive material due to its chemical stability, low cost and non-toxic properties, this study concentrated on TiO_2 nanoparticles in terms of the effect of different temperatures.

3.4 IR and optical property of TiO_2

The FTIR spectra of different TiO_2 powders after calcination are depicted from 4000 cm^{-1} to 700 cm^{-1} in Figure 5. It is well known that the variation modes of anatase skeletal O-Ti-

O bonds are observed in the range of 500 – 900 cm^{-1} , with a maximum at 474 cm^{-1} [6;7;8;9]. Commonly, the IR spectrum at about 3500 cm^{-1} , indicated the OH-stretching vibrations of free and hydrogen-bonded surface hydroxyl groups [6]. This suggests that water molecules are probably attached to TiO_2 surface with OH-stretching. The IR spectrum at around 1636 cm^{-1} , was also water-related functional group (H-O-H bending). However, at 100 °C calcination, three main spectra were found at 1360 cm^{-1} , 1425 cm^{-1} and 1220 cm^{-1} (Table 2).

Figure 4. Particle size distribution of (a) titanium oxide at 600 °C, (b) hematite and grattarolaite at 700 °C and (c) alumina at 1000 °C

Figure 5. FTIR spectra of TiO_2 powders after calcination at different temperatures

Table 2. FTIR analyses at 100 °C calcinations of Ti-salt floc

Figure 6 shows UV-visible absorption of P-25 and the different TiO_2 at different temperatures. P-25 photocatalyst absorbed the majority of UV light (less than 417 nm wavelength). However, the onset of the absorption spectrum of TiO_2 , calcination at 600 °C, 800 °C and 1000 °C appeared at 407 nm, 407 nm and 430 nm, respectively. The band gap was 3.18 eV (P-25), 3.05 eV (after 600 °C), 3.05 eV (after 800 °C) and 2.89 eV (after 1000 °C), respectively.

Figure 6. UV-visible absorption spectra of TiO_2 at P-25, 600, 800 and 1000 photocatalysts

3.5 SEM/EDX result

Table 3 shows atomic and weight percentage of the TiO₂ powders produced after different temperatures by EDX results. The carbon sources remained in all the ranges of different temperatures. The Ti, C, O and P atoms were mainly detected. In addition, many different trace elements (Si, Fe, Al, V, Ca, Na, Cr, Cl, S, Ni, and Br) were found.

Equation 1 shows the mass balance:

$$(\text{Ti input})_{\text{Theoretical}} - 0.6085 * (\text{TiO}_2\text{-output})_{\text{Theoretical}} = (\text{Ti input})_{\text{Experimental}} - 0.6085 * (\text{Particle production})_{\text{Experimental}} + \text{Impurity} \quad (1)$$

Here, 9.8 mg of Ti concentration was dosed and 17.86 mg of TiO₂ was experimentally recovered. Thus, the impurity was 1.78 mg in particles used in this study. The percentage of the impurity was 9.97%. However, in this study, four main elements are mainly handled, which are Ti, O, C and P. Here, the carbon compounds may be due to the remaining carbon from the settled organic floc, which was not volatile to carbon dioxide and water. The averaged elemental content at 100 °C, 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C in terms of Ti:O:C:P was 10.0:59.4:28.9:1.8, 11.2:61.4:26.0:1.36, 13.0:61.9:2.0:2.1, 13.2:64.6:19.8:2.4, 17.0:68.5:12.1:2.4 and 17.7:70.7:10.4:1.2, respectively.

Table 3. Atomic and weight (%) percentage of powders at different temperatures with $\pm 3.5\%$ of standard deviation (weight % (atomic %) of P25: Ti = 47.22 % (23.02%) and O = 52.78 % (76.98%))

3.6 Photocatalytic activity

The photocatalytic activity test with P-25 and TiO₂ produced from the Ti-salt sludge (600 °C, 800 °C and 1000 °C calcination) was investigated under irradiation of UV (Sankyo, F10T8BLB, three 10 W lamps) and visible light (Kumbo, FL10D, three 10W lamps) using the method of photodecomposition of gaseous acetaldehyde (Figure 7). As the BET surface area of TiO₂ after 600 °C (76.3 m²/g), 800 °C (5.96 m²/g) and 1000 °C (0.74 m²/g) was very different, the amount of TiO₂ used in the experiments was varied to achieve a constant amount of removal (20%) of acetaldehyde during TiO₂ adsorption following which it was irradiated with UV light. The concentration of acetaldehyde was measured by gas chromatography (Youngin, M600D). Photocatalytic removal of acetaldehyde with TiO₂ after 600 °C calcinations was higher than that with P-25 under UV irradiation. On the other hand, photocatalytic removal with TiO₂ after 800 °C and 1000 °C was lower than that with P-25. Although this figure is not shown, removal of acetaldehyde was small with all TiO₂ under visible light.

Figure 7. Variation of CH₃CHO concentration with irradiation time (initial concentration of CH₃CHO = 1000 mg/L; UV irradiation = black light three 10 W lamps; visible light irradiation = fluorescent light at 436 nm and a light power of 0.9 mW/cm²)

Conclusions

A detailed experimental study was conducted with Fe, Al and Ti salt coagulant to investigate their ability to remove organic matter, settling floc (sludge) and recycling sludge by calcination. The results led to the following conclusions:

1. Removals of organic matter by Fe, Al and Ti-salt flocculation at optimum concentration were 73%, 70% and 70%, respectively. The Fe floc settled quickly compared with Ti and Al flocs. The decantability decreased in the order of Fe floc > Ti > Al.

2. After calcination of the settled floc, Ti floc produced titanium oxide (TiO_2), Fe floc formed hematite (Fe_2O_3) and grattarolaite ($\text{Fe}_3(\text{PO}_4)\text{O}_3$ or Fe_3PO_7), and Al floc created alumina (Al_2O_3).

3. TiO_2 produced from the sludge consisted of the large amount of nano size particles. Hematite (Fe_2O_3) and grattarolaite ($\text{Fe}_3(\text{PO}_4)\text{O}_3$ or Fe_3PO_7) mostly included 2.75 μm micro size particles (40%). Alumina (Al_2O_3) consisted of micro size particles (40%).

4. The percentage of the impurity of TiO_2 produced by the settled floc was 9.97%. According to SEM/EDX elemental results, Ti, O, C and P elemental contents on/in TiO_2 were significantly changed at different temperatures. Photoactivity of TiO_2 at different temperatures was significantly varied. At 600 °C calcination, the photoactivity was very high. On the other hand, at 800 and 1000 °C, the photoactivity was marginal compared with that of P-25 photocatalyst.

This study has significant potentials to minimize a large amount of sludge from water treatment plants. The recovered sludge can be converted to useful metal oxides which are titanium dioxide, hematite and alumina. The significant quantities of metal oxides can be applied to environmental remediation, construction material and energy production.

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Table 1. Constituents of the synthetic wastewater

Table 2. FTIR analyses at 100 °C calcinations of Ti-salt floc

Table 3. Atomic and weight (%) percentage of powders at different temperatures with $\pm 3.5\%$ of standard deviation (weight % (atomic %)) of P25: Ti = 47.22 % (23.02%) and O = 52.78 % (76.98%)

Table 1. Constituents of the synthetic wastewater

Compounds	Concentration (mg/L)	Molecular weight (daltons)	Fraction by organic concentration
Beef extract	1.8	300, 100, 70	0.065
Peptone	2.7	34300, 100, 80	0.138
Humic acid	4.2	1500, 300	0.082
Tannic acid	4.2	6300	0.237
Sodium lignin sulfonate	2.4	12100	0.067
Sodium lauryle sulphate	0.94	34300	0.042
Arabic gum powder	4.7	900, 300	0.213
Arabic acid (polysaccharide)	5.0	38900	0.156
(NH ₄) ₂ SO ₄	7.1		0
K ₂ HPO ₄	7.0		0
NH ₄ HCO ₃	19.8		0
MgSO ₄ •7H ₂ O	0.71		0

Table 2. FTIR analyses at 100 °C calcinations of Ti-salt flocc

1220 cm ⁻¹	1) 1260 – 1180 cm ⁻¹ : alcohol (phenol-OH)
	2) 1240 – 1170 cm ⁻¹ : Amines (R ₂ -C-NH ₂)
1360 cm ⁻¹	Carbonate [6]
1425 cm ⁻¹	1) 1440 – 1405 cm ⁻¹ : phosphorus ((RR'R'')P=OP-CH ₂)
	2) 1440 – 1395 cm ⁻¹ : carbo acid (COOH)

Table 3. Atomic and weight (%) percentage of powders at different temperatures with $\pm 3.5\%$ of standard deviation (weight % (atomic %) of P25: Ti = 47.22 % (23.02%) and O = 52.78 % (76.98%))

		100 °C	200 °C	400 °C	600 °C	800 °C	1000 °C
C	Atomic (%)	28.85	26.04	23.00	19.75	12.08	10.38
	Weight (%)	18.83	16.69	14.00	11.99	7.15	5.88
O	Atomic (%)	59.39	61.37	61.92	64.62	68.50	70.66
	Weight (%)	51.64	52.39	50.21	52.26	54.03	53.31
P	Atomic (%)	1.77	1.36	2.08	2.41	2.40	1.23
	Weight (%)	2.98	2.24	3.27	3.78	3.67	1.80
Ti	Atomic (%)	9.99	11.23	13.00	13.21	17.02	17.72
	Weight (%)	25.99	28.68	31.55	31.97	40.16	40.00

* Trace elements found: Si (0.2%), Fe (0.02%), S (0.01%), Al (0.01), V, Ca, Na, Cr, Cl, Ni, and Br

Figure 1. Organic removal by different flocculations with synthetic wastewater (initial organic conc. = 11.5 mg/L)

Figure 2. Decantability ratio of different flocs

Figure 3. XRD images a) of the settled floc after TiCl_4 flocculation, b) of the settled floc after FeCl_3 flocculation (hematite (Fe_2O_3): 24° , 33° , 35° , 40.5° , 49.4° , 54° , 57.5° , 62.5° , 63.9° , 71.8° , 75.3° ; grattarolaite ($\text{Fe}_3(\text{PO}_4)_3$ or Fe_3PO_7): 18.2° , 28.9° , 36.8° , 38.9° , 43.3° , 45.3° , 45.6° , 49.2° , 55.1° , 56.7° , 59.7° , 60.1° , 61.5° , 64.7° , 70.4°) and the settled floc after $\text{Al}_2(\text{SO}_4)_3$ flocculation (Al_2O_3 : 32.3° , 34.6° , 36.9° , 39.0° , 45.0° , 46.3° , 60.8° , 67.8°) by calcination at different temperatures

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Figure 6. UV-visible absorption spectra of TiO_2 at P-25, 600, 800 and 1000 photocatalysts

Figure 7. Variation of CH_3CHO concentration with irradiation time (initial concentration of CH_3CHO = 1000 mg/L; UV irradiation = black light three 10 W lamps; visible light irradiation = fluorescent light at 436 nm and a light power of 0.9 mW/cm^2)

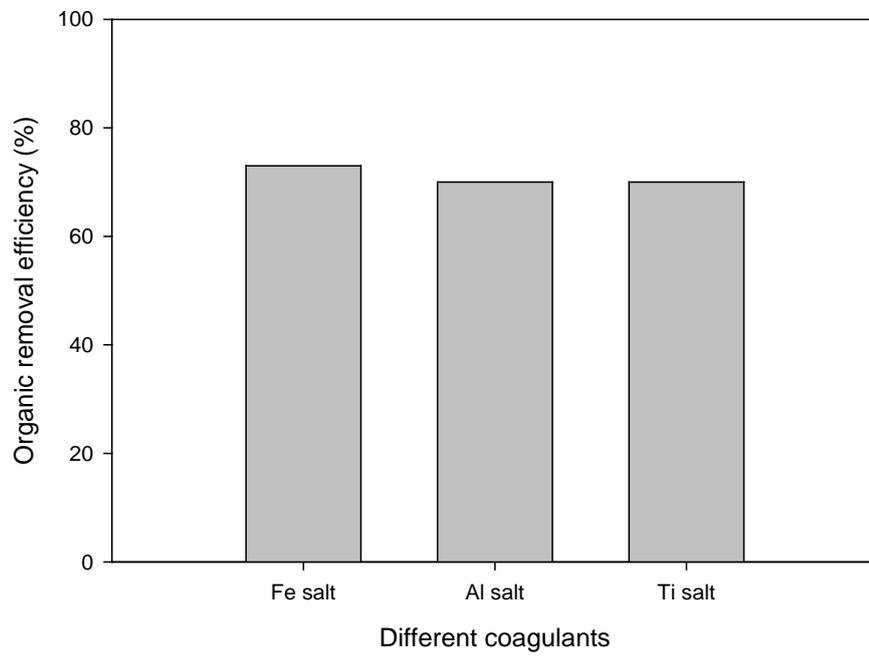


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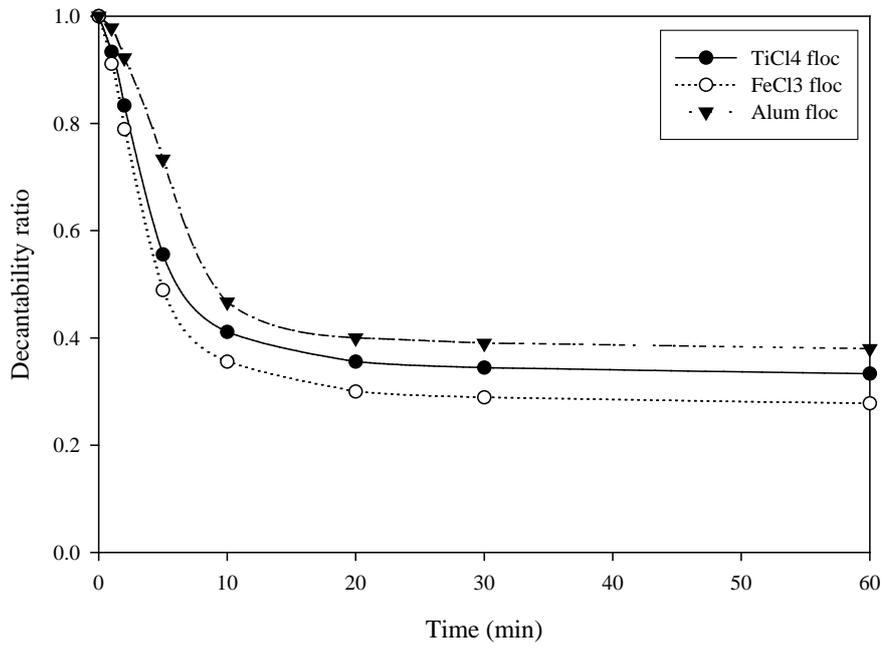


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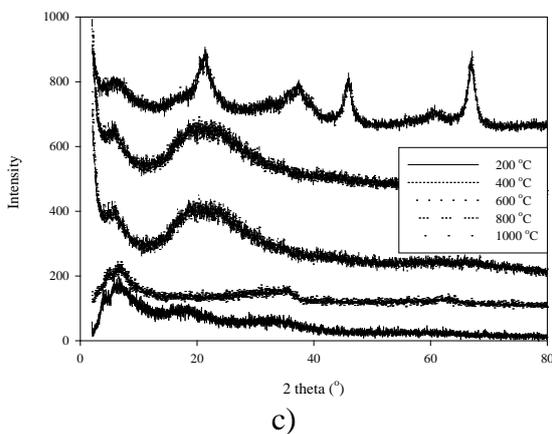
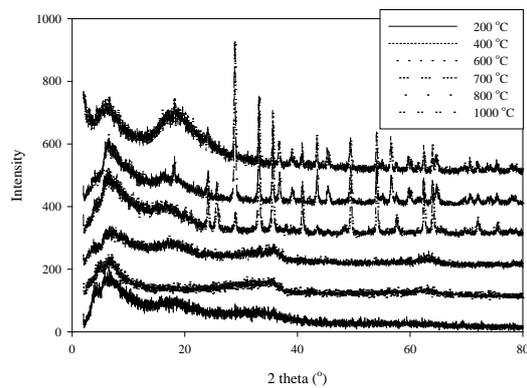
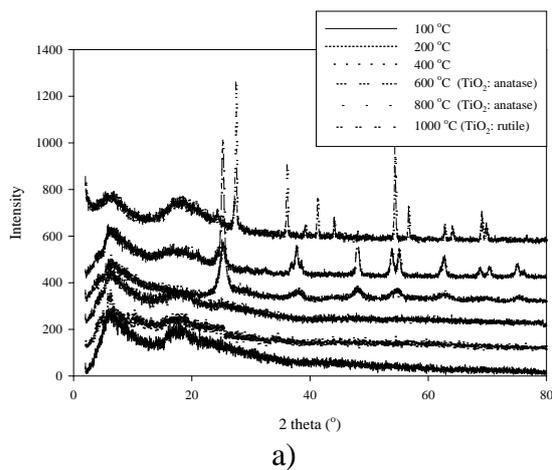
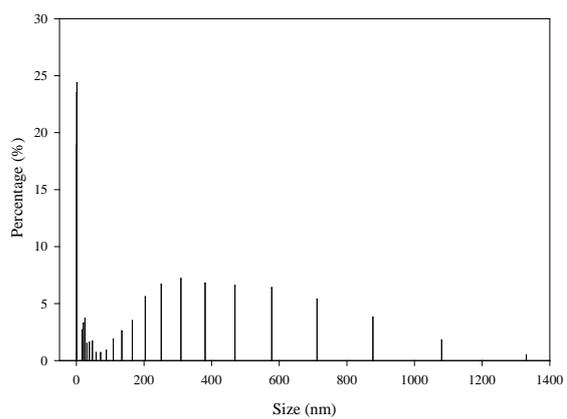
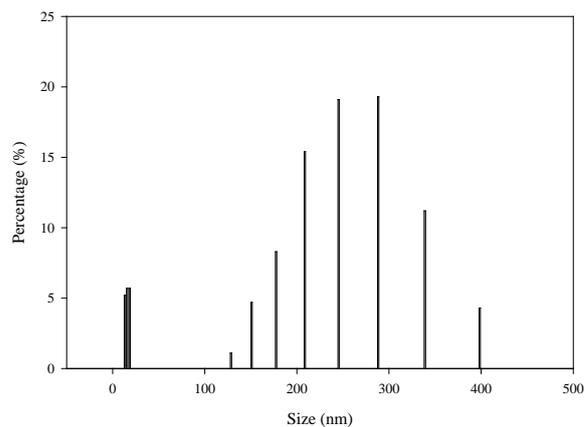


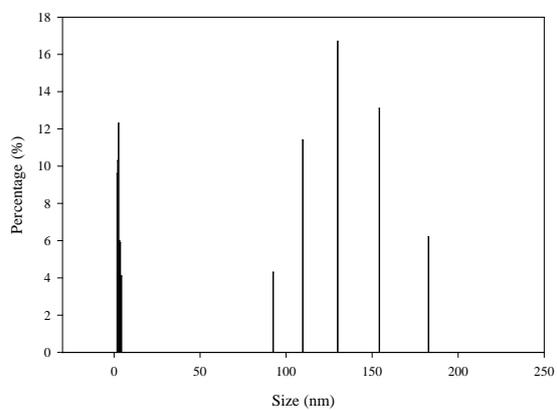
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a)



b)



c)

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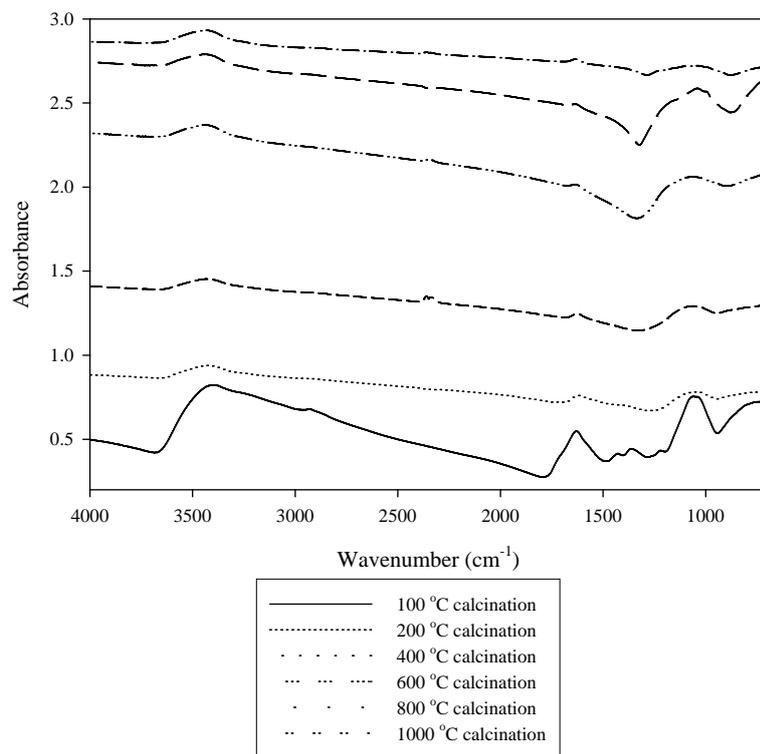


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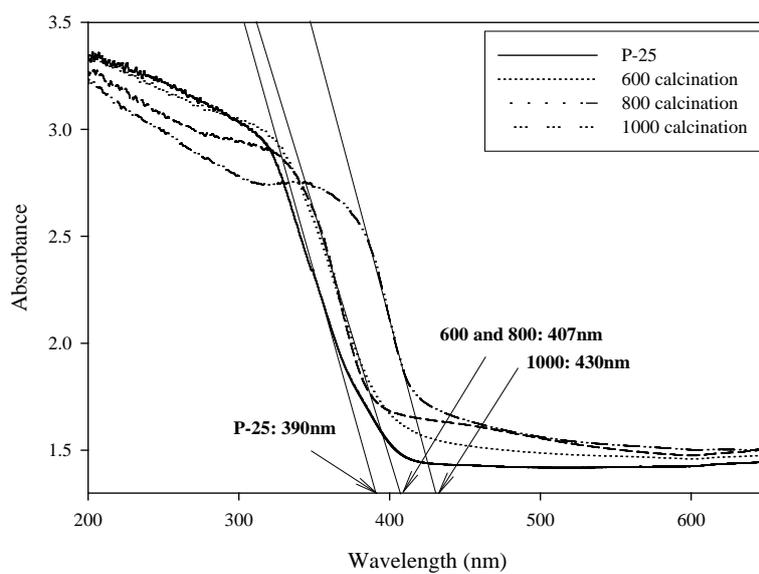


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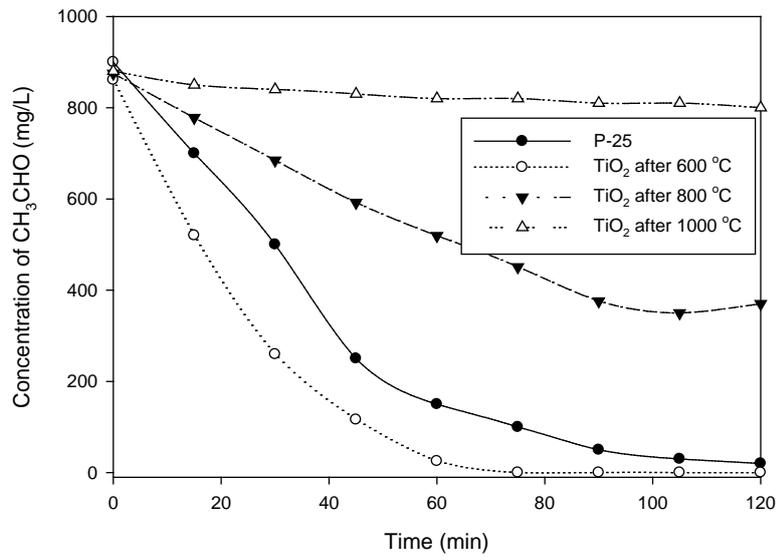


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