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Research Paper

Oil Palm Leaves as an In-situ Bio-silica Source in Sustainable Synthesis of $V_2O_5-SiO_2$

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Abstract
Using ammonium vanadate and natural silica from oil palm leaves in situ at 900°C is a sustainable synthesis method for producing $V_2O_5-SiO_2$ mixed oxides in the form of a brownish powder. Therefore, this study aims to investigate a more environmentally friendly alternative to synthesizing $V_2O_5-SiO_2$ using oil palm leaves, a by-product from oil palm farming. The XRD analysis of the reaction products showed specific V_2O_5 peaks and broadened peaks, indicating the presence of amorphous silica. The Fourier transform infrared (FTIR) analysis, which revealed the presence of Si−O−Si and Si−O−V functional groups, also supported the characteristic assessment. In addition, X-ray fluorescence (XRF) analysis showed that V_2O_5 (46.70 mass%) and SiO_2 (52.60 mass%) were present, along with small amounts of other possible metal oxides, such as P_2O_5, K_2O, CaO, Fe_2O_3, Al_2O_3, and PdO.

Keywords
V_2O_5, Biosilica, Oil Palm Leaves, Vanadium Pentaoxide, Sustainable

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1. INTRODUCTION

Inorganic composites or mixed oxides are increasingly recognized for their superior properties compared to individual compounds. Previous reports have detailed some research on the preparation of composites using silica (SiO_2) as a partner (Wardhani et al., 2023; Sinaga and Lesbani, 2017). An example of a composite that has attracted attention is the $V_2O_5-SiO_2$. The main focus is on synthesizing alternatives to achieve specific characteristics and exploring their potential applications. Several studies have developed some techniques to synthesize $V_2O_5-SiO_2$ composites such as sol-gel, combustion, and hydrothermal methods. For example, $V_2O_5-SiO_2$ composite was synthesized by mixing V_2O_5 solids with SiO_2 suspensions in chloroform. The product obtained was used as a catalyst to prepare 5-aminopyrazole derivatives (Khatab et al., 2019) or to oxidize methane (Bostan et al., 2002). Synthetic precursors, such as tetraethoxysilane (TEOS), are known as commercially available sources of silica. $V_2O_5-SiO_2$ was produced using a sol-gel method from TEOS, and the product catalyzed the oxidative dehydrogenation of n-butane compounds (Murgia et al., 2006). Other studies performed a similar method and used the resulting product in the catalytic aromatization of 1,4-dihydropyridine compounds (Farzaneh et al., 2012). In addition, $Na_2SiO_3.9H_2O$ has been reported to be a silicon source, while NH_4VO_3 serves as a vanadium source (Wang et al., 2017). Several studies have shown the role of Na_2SiO_3 as a silica source, with the resulting composite product serving as an anode for liquid lithium batteries (Zhang et al., 2016). Some studies have also focused on the phases that occur during the $V_2O_5-SiO_2$ composite formation process at certain temperatures. Due to the melting of V_2O_5 at 680°C, the current study shows the existence of the composite phase equilibrium (Feng et al., 2020). Another method that was developed as an alternative is flame spray pyrolysis (FSP) due to the potential use of composites as cathode materials for rechargeable mg-ion batteries (Tanaka et al., 1989).

In a previous study, the sodium ions (Na^+)-modified V_2O_5/SiO_2 were modified and applied as photocatalysts for the oxidation of 2-propanol and propene (Colpini et al., 2013). V_2O_5/SiO_2 synthesis using the sol-gel method was also developed for NO reduction in the catalytic version (Tavassoli et al., 2016). Recent studies also focus on varying the V_2O_5/SiO_2 synthesis based on the type of synthetic precursor. The results showed that the catalysts were effective for oxidizing SO_2 to SO_3 (Mostafa and Khatab, 2018). The direct preparation of silica-supported V_2O_5, which involved mixing 2 metal oxides in a slurry form and then evaporating the used solvent. The prepared material served as a catalyst in multicomponent reactions, resulting in the production of 4H-pyran derivatives (Rohilla and Kumari, 2015). The coprecipitation technique was employed to produce binary oxide nanocomposites of vanadium pentoxide (V_2O_5) and silica (SiO_2). Metallic chlorides were used as precursors, which were in situ formed from V_2O_5 and HCl in the reaction mixture. The composites were subsequently annealed at a variety of temperatures for 2 hours (Eurov et al., 2023). The synthesis of V_2O_5/SiO_2 through hydrolysis using ammonium vanadate as a precursor for V_2O_5, and tetraethoxysilane for SiO_2 has been widely reported. At ambient temperature, this compound exhibits luminescence (Bokarev et al., 2024). The use of $V_2O_5-SiO_2$ in wound care and treating skin cancer cells with cream is also a very interesting development (Ali and Drea, 2023).

Many studies have examined the use of bio-silica for making advanced materials used in labs and farming. For instance, Bi_2O_3 and Bi_2SiO_5 have been created using bismuth nitrate along with different silica sources, such as rice husk, rice straw, and even mineral-based silica (Arefieva et al., 2023). Recently, researchers have also managed to get bio-silica from rice straw digestates and raw rice straw that had been treated with agro-industrial waste products, such as corn-steep liquor and cassava-steep wastewater (Ekwenna et al., 2023). The studies showed that it was possible to get silica (SiO_2) from rice husk and then coat it with magnetic nanoparticles (MNPs), which helps in removing phenol from solutions effectively (Wee et al., 2023). Also, using high heat in an open-air furnace, silica has been taken from bagasse and corn cobs, and this silica was then used for germinating *E. sativa* seeds (Goswami and Mathur, 2022).

Although there is a report on the green way of making SiO_2/V_2O_5 using rice husk ash, it still involved getting silica first and then making the composite through the sol-gel method. In addition, there is clear potential, the method for making V_2O_5–SiO_2 composites using natural silica sources is still not widely used (Khatab et al., 2019; Ali and Drea, 2023). This creates an opportunity to look into other approaches that could be more eco-friendly and also more cost-effective (Yudha et al., 2020). Some studies have already explored how synthetic silica affects the environment and what advantages bio-silica might offer. The outcomes show that the benefits can really vary depending on the method used, the region, and the type of raw material involved (Errington et al., 2023). Since natural resources are already available, exploring their use could be useful for industrial application. The presence of silica compounds has been confirmed from burning palm leaves, which are utilized for various purposes (Onoja et al., 2018b; Onoja et al., 2018a). Therefore, this study aims to investigate a more environmentally friendly alternative to synthesizing $V_2O_5-SiO_2$, which utilizes oil palm leaves, a waste product from oil palm farming. The pruning process of the palm oil farming system yields palm leaves as a by-product, which is crucial for achieving optimal palm fruit production.

2. EXPERIMENTAL SECTION

2.1 Materials

This study made use of materials, such as hydrochloric acid and ammonium vanadate (Merck, Germany), demineralized (Akuadm) and distilled water, and oil palm leaves. There was also use of many kinds of glassware from the chemistry lab, such as beaker glass, erlenmeyer flasks, a magnetic bar, volumetric pipette, and other needed glass tools that were helpful.

2.2 Methods

About 100 grams of palm leaf powder was put in a 1000 mL beaker. Then, 500 mL of 10% HCl was poured into the same beaker that had the powder, and it was stirred on a magnetic hotplate stirrer. The hotplate got turned on and was left running until the heat went up to around 80-90°C. The stirring and heating kept going at that level for roughly 3 hours or so. After that, it was left to cool down to room temperature and stayed like that for 15 hours (Onoja et al., 2018a). After waiting, the HCl was poured out, and the solid part, the palm powder, was washed using distilled water. Washing was done a few times till the water became about pH 6 to 7 (Wang et al., 2003). After this, the treated black powder from the palm leaf was put out in the sun to dry, for 8 hours each day, two days in a row (making 16 hours total).

Ammonium vanadate was used to make V_2O_5, and the method followed a procedure that was proposed by Eurov et al. (2023). Ammonium vanadate (1.697 grams), was added into 50 mL of demineralized water that was already heated to about 80°C. This was done using a hotplate that had a magnetic stirrer, and it was used to mix the solution properly. Approximately 5 grams of palm leaf powder that had been cleaned of minerals as described in the previous stage was added to the hot solution. This was then stirred to ensure that the mixture coalesced and formed a semi-solid material. The mixture was allowed to stand for 24 hours for aging, and then the crucible was placed in the furnace, which was heated to 900°C, and the reaction was conducted for 5 hours. After cooling to room temperature, the resulting material was ready to be characterized.

The product material was analyzed for its phase characteristics using X-ray diffraction (XRD) analysis (Rigaku Miniflex 600). X-ray fluorescence (XRF) (Rigaku NEX DE Series) was performed for elemental analysis of the materials. The material was also analyzed using a Fourier transform infrared (FTIR) spectrophotometer (Bruker Alpha II) and scanning electron microscopy (SEM) (FEI, Inspect-S50). Furthermore, data from the measurements using these instruments were collected and analyzed to gain an understanding of the characteristics of the resulting material.

3. RESULTS AND DISCUSSION

The study process commenced with the collection of oil palm leaf as depicted in Figure 1. In Figure 1(a), the fronds of oil palm leaf were removed from the leaf, separated from the lid, and then cut into small pieces using scissors to obtain the material shown in Figure 1(b), which was crushed in a blender and obtained as palm leaf powder, as shown in Figure 1(c). The washing of palm leaf powder using HCl as described in the procedure, aimed to remove possible metal ions in oil palm leaf

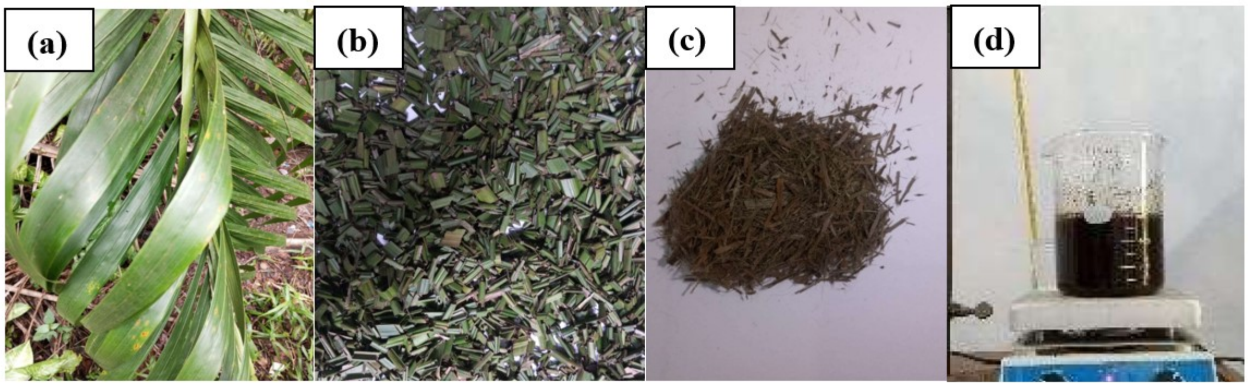

Figure 1. The Process Included Processing Oil Palm Leaf from the Field to the Washing Stage, using a Hydrochloric Acid Solution. (a) Oil Palm Leaf (Origin); (b) Coconut Leaf Flakes; (c) Un-treated Oil Palm Leaf Powder; (d) Treatment of Oil Palm Leaf Powder Using 10% HCl Solution

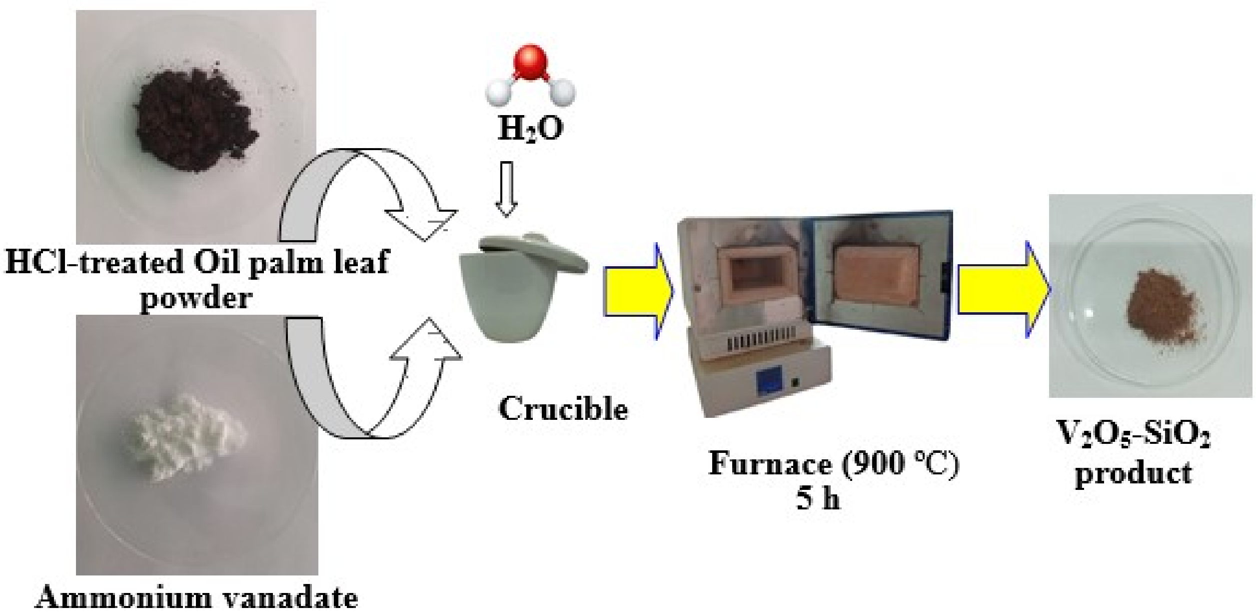

Figure 2. The Schematic Processes of Preparing $V_2O_5-SiO_2$ Using HCl-Treated Oil Palm Leaf Powder and Ammonium Vanadate in a Furnace at 900°C for 5 Hours

that were not needed in this study. This procedure was commonly used in the removal of certain metal ions (Vercruysse et al., 2023; Tabish et al., 2024). Therefore, it was expected to be less disruptive in subsequent reactions. In this study, the analysis of metal ion content determination before and after washing was not performed. SiO_2 content obtained directly from oil palm leaf generally had a small yield, but in this study, the material used was treated oil palm leaf using 10% HCl accompanied by heating. Furthermore, the treatment using HCl assisted by heating greatly reduced the impurities present in oil palm leaf powder either inorganic minerals or some organic compounds that dissolved in the HCl solution. This was indicated by the change in color of HCl solution from clear to blackish after the washing process using HCl, which was assisted by heating at the temperature of 90°C. In addition, heating one gram of the current HCl-treated oil palm leaf produced ±9% silica content.

As shown in Figure 2, HCl-treated oil palm leaf powder material was used as a precursor for the synthesis of $V_2O_5-SiO_2$ mixed oxides by mixing with ammonium vanadate. The reaction was started by making the mixture in a crucible and adding demineralized water to make it paste, which was left for 24 hours to allow the mixture to interact with each other. Subsequently, the semi-solid mixture obtained was heated at 900°C for 5 hours, and the product obtained was a brownish solid.

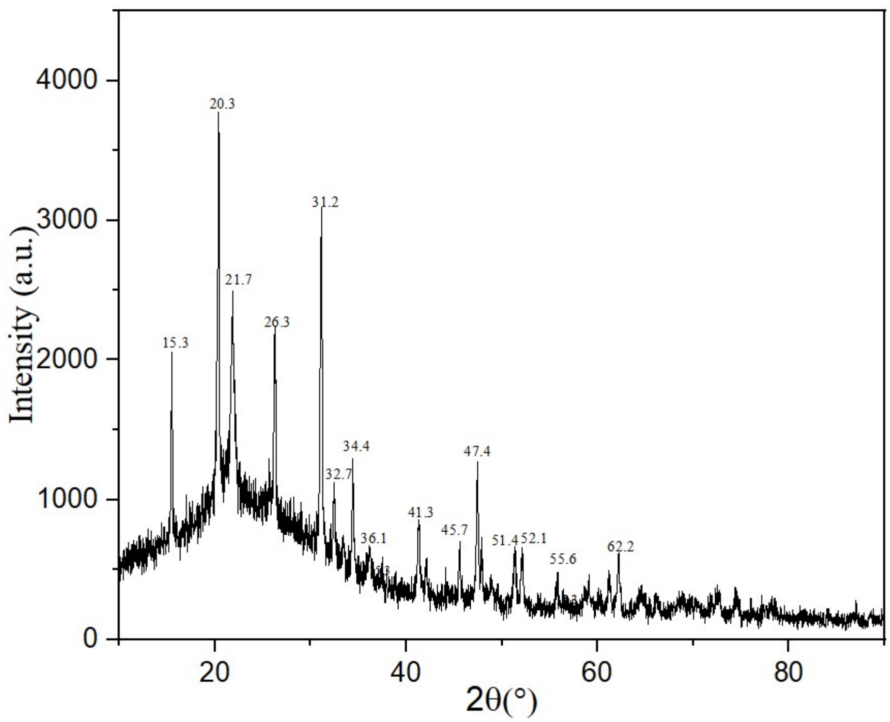

Figure 3. X-ray Diffraction Pattern of the As-Prepared $V_2O_5-SiO_2$

Figure 3 showed the XRD analysis results of the obtained powder, showing specific peaks corresponding to V_2O_5 and amorphous SiO_2. Some special peaks were observed at 2θ: 15.3°, 20.3°, 21.7°, 26.3°, 31.2°, 32.7°, 34.4°, 41.3°, 45.7°, and 47.4°. This was consistent with JCPDS card No. 00-041-1426 for orthorhombic α-V_2O_5 which was indicated by peaks at 15.5°, 20.31°, 20.4°, 21.8°, 26.3°, and 31.1° and 41.12° (Khatab et al., 2019) and no tetragonal phase of β-V_2O_5 detected. However, there was a widened peak appearance at 2θ from 35° to 10° in between V_2O_5 peaks, which showed SiO_2 peaks and was consistent with JCPDS card number 29-0085 for amorphous SiO_2 (Bostan et al., 2002; Yudha et al., 2020; Badri et al., 2020). Based on the full width at half maximum (FWHM) of a diffraction peak of V_2O_5 and calculated using the Scherrer formula, the crystal size of solid particles produced in this study was 17.9 nm. To obtain a morphological description of a solid product obtained in the form of $V_2O_5-SiO_2$, an analysis using SEM was carried out and the results were shown in Figure 4.

As shown in Figure 4(a), the morphology of solid materials was observed as a collection of overlapping spherical clusters. This was proven when the microscope magnification used was 20,000× Figure 4(b), which clearly showed the spheres of solid particles enveloped by crystalline hairs that were probably derived from SiO_2 produced in the experiments. Based on the analysis of the spheres, all were generally below 5 μM with an average value of about 3.5 μM.

FTIR analysis results shown in Figure 5 revealed that wavenumbers 3437 cm^{-1} and 1631 cm^{-1} denoted O−H functional groups, while wave numbers 2928 cm^{-1} and 2865 cm^{-1} signified C−H functional groups. Wave numbers at 1108 cm^{-1}, 921 cm^{-1}, and 804 cm^{-1} corresponded to Si−O−Si and Si−O−V functional groups. This was consistent with the results of FTIR analysis of V_2O_5/SiO_2 produced from vanadyl-acetylacetonate and tetraethyl-orthosilicate in refluxing MeOH (Farzaneh et al., 2012), showing the presence of Si−O−Si and Si−O−V vibrational peaks at wave numbers 1110 cm^{-1}, 934 cm^{-1}, 806 cm^{-1}, and 471 cm^{-1} . Other FTIR study results also demonstrated the presence of Si−O−Si symmetry bond vibrations indicated by strong absorption bands at 475 cm^{-1}, 568 cm^{-1}, 808 cm^{-1}, 965 cm^{-1}, and 1091 cm^{-1} and supported by the formation of

Table 1. Metal Oxide Content in the As-prepared Sample Analyzed with XRF

Entry	Metal Oxide	Intensity (kcps)	Mass (%)
1	Al_2O_3	0.44	0.09
2	SiO_2	300.22	52.60
3	P_2O_5	0.55	0.05
4	K_2O	2.99	0.24
5	CaO	1.97	0.17
6	V_2O_5	275.32	46.70
7	Fe_2O_3	0.50	0.03
8	PdO	8.49	0.07

Note: The mass (%) of metal oxide was calculated from the intensities obtained for each element (corresponding to Figure 5) using the formula installed in the software application inside the XRF instrument

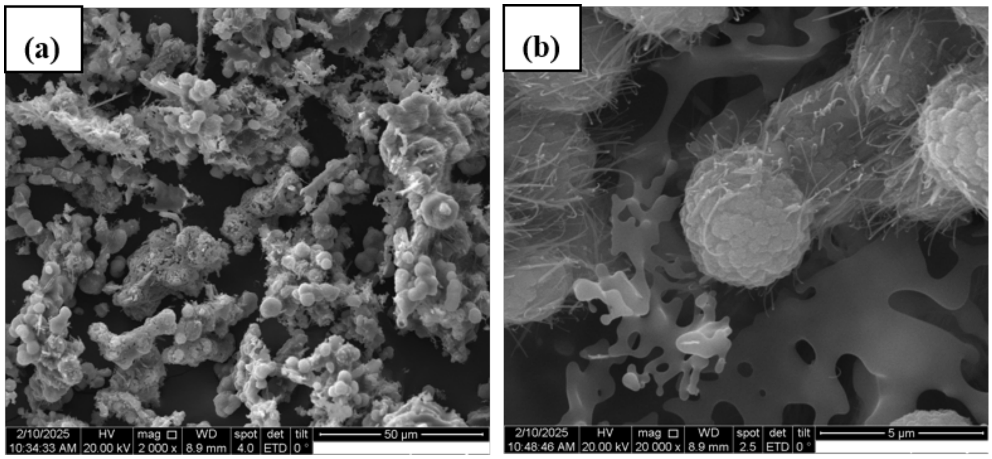

Figure 4. Scanning Electron Microscopy (SEM) Pattern of the As-Prepared $V_2O_5-SiO_2$ with a Magnification of (a) 2000× (b) 20000×

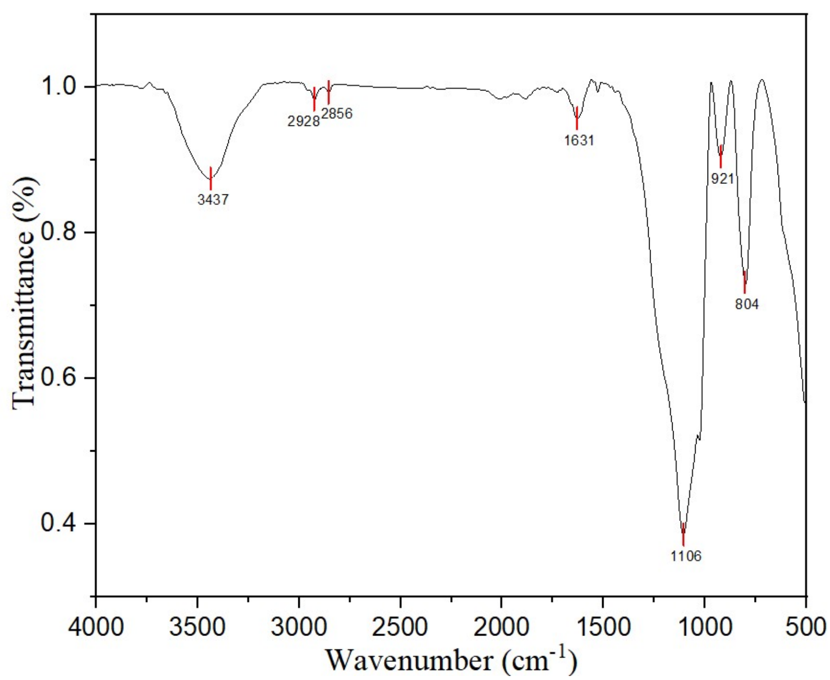

Figure 5. FTIR Spectrum Pattern of the As-Prepared $V_2O_5-SiO_2$

poly-vanadyl species at 960-990 cm^{-1} (Rohilla and Kumari, 2015; Ohde et al., 2008).

The results obtained from the FTIR test in this study, along with studies, suggest that the material made by this method turned into a $V_2O_5-SiO_2$ mixed oxide. This might be because of some vibrational bands that appeared, probably from Si−O−Si and Si−O−V groups. However, using this analysis, it had been difficult to reach a definitive conclusion regarding the resulting composite formation compared to the usual mixing of metal oxide V_2O_5 and SiO_2. Previous studies showed that it was possible to strengthen predictions about composite formation when supported by more accurate analysis, which could help in understanding surface composition, bonding, and element distribution (Zhang et al., 2023).

The synthesized solid material was analyzed to determine the composition of metal oxides that could be present in it, and the results were shown in Figure 6 (XRF spectrum) and Table 1 (calculated mass (%) of the metal oxide contents).

The results were V_2O_5 (46.70 mass%) and SiO_2 (52.60 mass%) with the other possible metal oxides in very minor amounts. These supported XRD analysis that showed the presence of both major oxide compounds in the as-prepared material. The current analysis results also suggested that the use of ammonium vanadate was suitable as a precursor for in-situ syn-

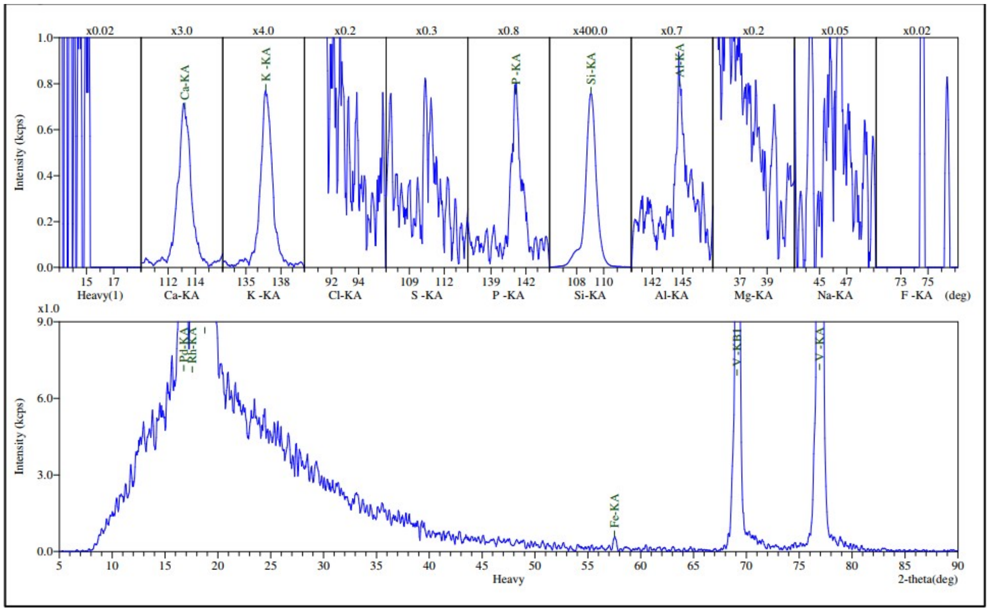

Figure 6. XRF Spectrum of the As-Prepared $V_2O_5-SiO_2$ (The Continuous Lines Were Composed of Straight Lines Between the Individual Channel Values)

thesis of $V_2O_5-SiO_2$ using combustion methods of oil palm leaf. Moreover, the results showed that the ratio between V_2O_5 and SiO_2 was approximately 1:1. Some further reports focusing on the beneficiary of V_2O_5/SiO_2 were observed, such as oxovanadium complexes which altered the composites and provided unique properties and catalytic applications (Jayasayee et al., 2016).

Based on the studies that had been carried out, the utilization of palm leaf by-products as an alternative source of SiO_2 was a companion to other SiO_2 sources such as rice husk and also commercially available synthetic SiO_2 sources. The advantage of this natural SiO_2 source, was the abundance of by-products, ensuring that it was easily accessible for the development of other studies. Furthermore, the utilization of palm leaf also had an impact on the added value of oil palm industry by-products.

4. CONCLUSIONS

In conclusion, $V_2O_5-SiO_2$ mixed oxides were synthesized from oil palm leaf using an in-situ decomposition-oxidation method at a high temperature (900°C) in an open-air furnace. The reaction proceeded smoothly, yielding the desired product. Furthermore, V_2O_5 obtained was alpha-V_2O_5 (orthorhombic), and amorphous SiO_2, which was another oxide in the current mixed oxides. The successful synthesis by using this natural resource could encourage efficiency and atomic economy in inorganic synthesis.

5. ACKNOWLEDGEMENT

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