Chapter

Nanocomposite Material Synthesized Via Horizontal Vapor Phase Growth Technique: Evaluation and Application Perspective

Muhammad Akhsin Muflikhun, Rahmad Kuncoro Adi and Gil Nonato C. Santos

Abstract

The synthesis of nanomaterials has been reported by many researchers using different methods. One of the methods that can be used with perfect pureness and have less pollution in the synthesized materials results is the vapor phase growth technique (VPGT). Several types of nano shapes materials were reported such as nanoparticles, nanorods, nano triangular, nanosphere, and nanocrystal. The synthesis method has a fundamental process where the nanomaterials evaporated and condensed based on the temperature difference. There are three important variables, i.e., stochiometric ratio of source materials, temperature and baking time. The synthesis was occured in the quartz tube and sealed in the vacuum condition. This create the material was synthesis in pure and isolated conditions. The application of the nanomaterials synthesized via Horizontal Vapor Phase Growth (HVPG) can be implemented in anti-pathogen, anti-bacterial, gas sensing and coating applications.

Keywords: HVPG, synthesis nanomaterials, phase transition, anti-bacterial, coating, sensing applications

1. Introduction

Recently, nanotechnologies and nanoscience have raised high hopes for a new potential industry revolution [1]. They produced materials of various types at nanoscales [2]. Nanotechnology is commonly used in many applications such as in industrial, medical, agriculture, aerospace, energy, automotive, and food. Many researchers have conducted to explore the field of nanotechnology. They focus to obtain to get the best nanomaterials with optimal mechanical and physical properties [3–5].

Nanomaterials are a wide class of materials that have a range of the dimension 1 nm-100 nm at least and they are made from nanoparticles [2, 6, 7]. There are several methods to synthesis a nanomaterial that was developed by many researchers.

There is chemical reduction [8, 9], chemical vapor deposition [10], photochemical [11], electrochemical [12], green synthesis [13], photochemical [11], Horizontal Vapor Phase Growth (HPVG) [14, 15], photochemical [11], microwave [16], sol–gel [17], and sonochemical [18]. One of the methods that successfully synthesized nanocomposite materials is the HPVG technique. HVPG was proven to be used to develop and able to produce material with various dimensional of nanostructures like Fe_2O_3 [19], Ln_2O_3 [20], Ag-TiO_2 and SnO_2 [21]. This technique offers some advantages like economical, reliable method, and less source material with high purity [22, 23]. HVPG technique is also capable to create nanomaterials with various shapes such as nanoparticles, nanotubes, nanorods, and triangular nanomaterials [14, 24–26]. They were evaluated to investigate the structure, chemical composition, hardness, and morphological behavior of the nanocomposite material [27]. Previous research about evaluated nanocomposite material synthesized with the HVPG technique is present in **Table 1**.

The applications of HVPG techniques on synthesized nanomaterials were varies among the different sectors. Tibayan et al. [23, 29] used HVPG to synthesized Ag/SnO2 nanocomposite materials that can be used in coating applications. The characterization process used UV filtering analysis to evaluate the UV blocking. The results showed that the UV can be blocked efficiently. Moreover, DFT analysis using An and Ag as the sample material also showed that the entire spectrum of UV light can be absorbed with the model. The summary of the previous research about synthesized nanomaterials using the HVPG techniques can be seen in **Table 2**.

Based on the explanation above, nanomaterials were synthesized by the HVPG technique and evaluated using several tests such as Scanning Electron Microscope (SEM), Energy-Dispersive X-ray Spectroscopy (EDX), X-Ray Diffraction Analysis (XRD), Density-Functional Theory (DFT), Applied Behavior Analysis (ABA),

Source	Method to evaluate	Function
Muflikhun et al. [28]	SEM	To determine the image and measure the synthesized nanomaterial
	EDX	To determine the composition of the material
	AFM	To determine the 3D surface roughness of nanocomposite material
Tibayan et al. [29]	SEM	To determine the image and measure the zone in synthesized nanomaterial
	DFT	To determine the atomic geometry and electronic properties the nanocomposite material
	ABA	To determine the metabolic activity in the material
Motlagh et al. [30]	SEM	To investigate the dispersion of nanoparticles
	AFM	To investigate the surface roughness of the coating
	Elcometer Motorized Scratcher	To determine the scratch resistance
	Sheen Pendulum Hardness (707 KP)	To determine the surface hardness
	Micro-Tri Gloss Meter (BYK-Gardner)	To determine the gloss coating
Reyes and Santos [31]	SEM	To determine the image of nanomaterial
	EDX	To determine the composition of the material

Table 1.
Nanocomposite materials evaluation on previous research.

Source	Years	Nanomaterials name
Muflikhun et al. [32]	2019	Synthetic Silver (Ag/TiO_2)
Shimizu et al. [24]	2016	Skewered phthalocyanine $[Fe^{III}(Pc)(SCN)]_n$
Quitaneg and Santos [33]	2011	Cadnium Selenide (CdSe)
Reyes and Santos [31]	2011	Tin Dioxide (SnO_2)
Buot and Reyes [34]	2012	Carbon-silver (C/Ag)
Muflikhun et al. [15]	2017	Silver-graphene (Ag/Ge)

Table 2.
Previous research of synthesized nanomaterials using HVPG technique.

and Atomic Force Microscope (AFM), representing advances in the development of nanomaterials. The above discussion can also be used for further research in developing large-scale nanomaterials that can be applied in the industrial world.

2. The HVPG method

Detailed of the HVPG method are described in the study of Muflikhun et al. [21]. The step-by-step synthesis of nanomaterials is shown in **Figure 1**. It can be explained in the sub chapter 2.1 to 2.3 where all process was followed.

2.1 Preparation

Before the source material is inserted into the tube, the materials should be measured in the high precision weight scale to get the exact amount of material with a total weight of 35 mg. If the sample was composed of more than 1 sample, it should be divided into the individual sample. For example, the target synthesis was Ag/TiO_2 nanocomposite materials. The source materials that need to be prepared were Ag powder 17.5 mg and TiO_2 powder 17.5 mg. the combination should be equal to reach optimum results.

After material measurement, the next step was tube preparation. The tube was purchased from the general market nearby the university. The tube was originally used as the heater tube used in the toaster or heating unit. The tube before used as the place to grow nanomaterials, it should be washed in the ultrasonicator to remove the pollutant and impurity. After washed, the tube then dried until the water was evaporated. The one-side seal was applied to ensure the material that poured into the tube is trapped in the base. The one edge seal was also the final point of preparation where the specimen can be poured into the tube and then placed in the Thermionic High Vacuum System (THVS).

2.2 Sealing

Sample sealing is another important aspect to ensure the material is sealed properly. The tube before final sealing was placed vertically where the material was at the bottom. The upper tube is then joined with the pipe for the vacuum process. The process was occurred at a very low pressure (10 $Torr^{-6}$ Torr). The low pressure has purposes pushing the melting point of the materials. Sealing is also a critical process when the sealing is not proper, the leak will allow outer air to go inside the tube and the vacuum process failed. The sealing process also needs special treatment due to the used blow torch that used LPG and O_2 gas.

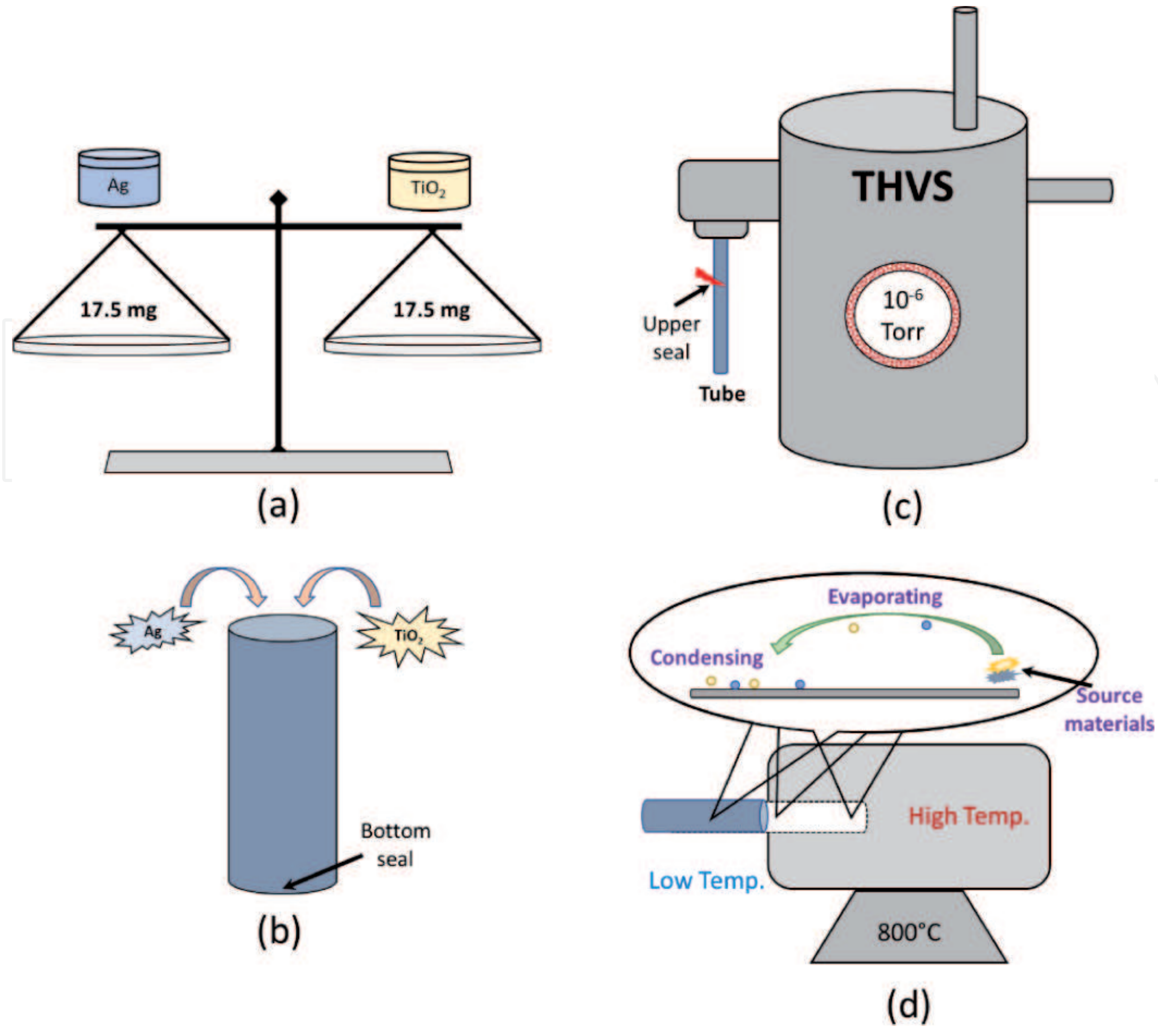

Figure 1.
(a) Material measurement, (b) pouring the materials into the tube, (c) final sealing process in vacuum condition, (d) baking process.

2.3 Baking

The HVPG method to synthesis nanomaterial is based on the temperature difference between 2 points. It is shown that the material that received heat in a continuous-time, the phase of material will change as follow:

From **Figure 2**, it is shown that the phase of the material will be melt and then evaporate after the critical point was overlapped. This phase transition occurred due to high temperatures in the furnace. The material then condenses and then become solid after moving from high temperature (inside the tube) to low temperature (outside the tube).

3. The study results and discussion

After cured with designated temperature and time, the nanomaterial inside the tube can be characterized and evaluated using the electron microscope. Since the unique phase transition of the materials that flow inside the tube, the nanomaterial growth inside the tube is also fascinating in terms of shape and size. The study from Muflikhun et al. [15] showed that flower-like and jellyfish-like nano-silver was successfully grown from the combination of Ag/Ge materials. The silver was grown on the graphene multilayer as shown in **Figure 3**. The growth variable in HVPG for the Ag/Ge was 1200°C and 6 hours baking time.

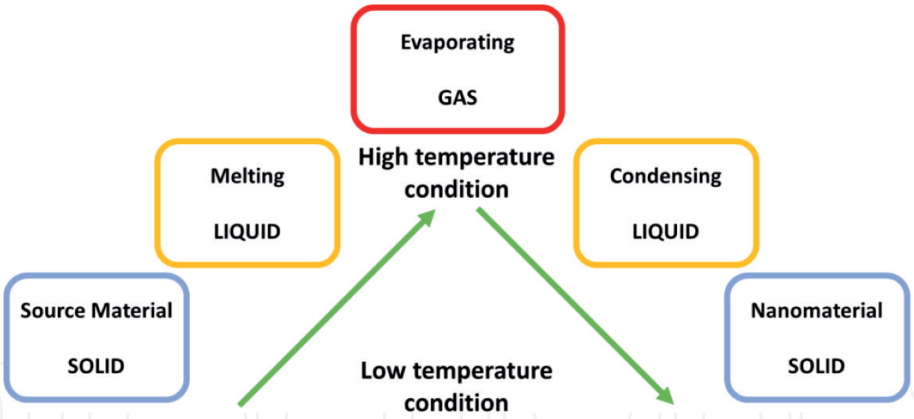

Figure 2.
Phase transition of materials in HVPG [21].

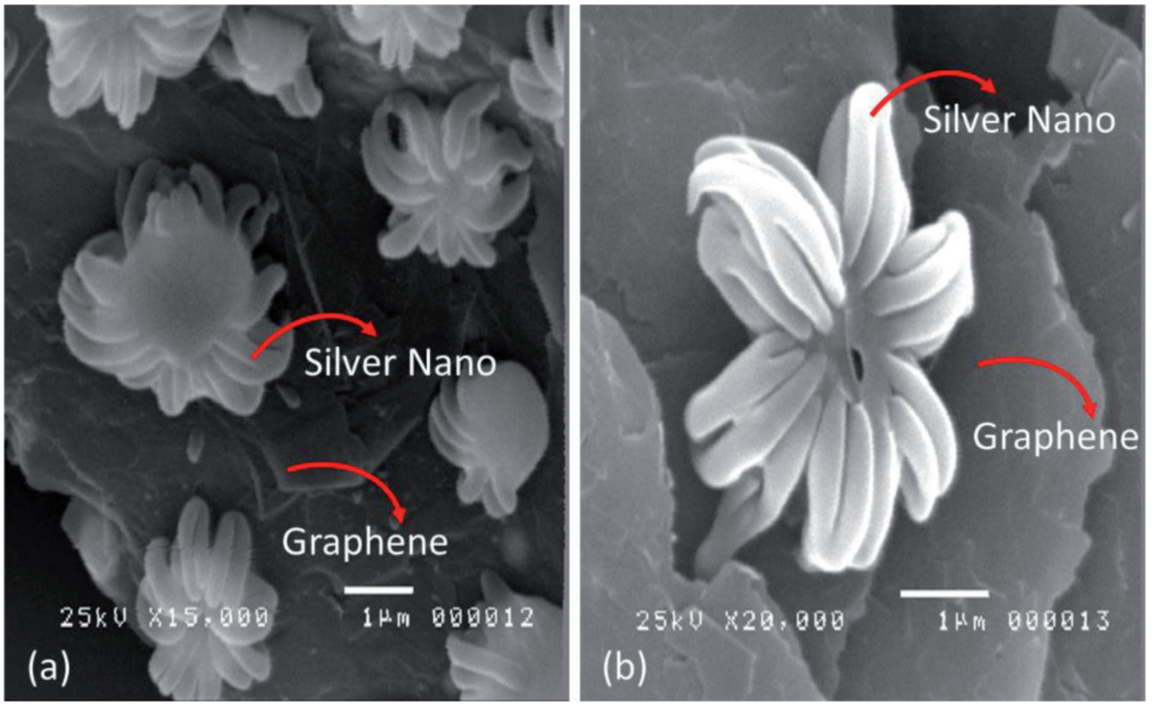

Figure 3.
(a) Jellyfish-like silver nanomaterial, (b) flower-like silver nanomaterial.

Another study conducted by Bernardino and Santos [35] shows that HVPG can be used to synthesis Gallium Oxide/Tin Oxide Nanostructures. The synthesis was used variable temperature 1200°C and 6 hours baking time. The results showed that different nano shape was grown such as nanowire, nano particles, and nano crystal.

There are 3 main variables to grow nanomaterial using the HVPG technique: time, temperature, and the weight ratio of the source material. During the baking process in the furnace, time and temperature play an important role to develop the shape of nanomaterials in the results. These two variables have been reported by Muflikhun et al. [28, 32]. The time was set to 4 hours, 6 hours, and 8 hours and the temperature was set to 800°C, 1000°C, and 1200°C. By using these 6 parameters, the 27 combinations of time and temperature can be achieved. The results of these results showed that the more different nanomaterials shape was successfully synthesized with different shapes of nanomaterials as reported below (**Table 3**).

No.	Temperature	Baking time	Zone	Material shape and diameter
1	800°C	4 Hours	1	Nanoparticles
2			2	Micro particles
3			3	Micro particles
4		6 Hours	1	Nanospheres, Nanoparticles
5			2	Nanoparticles
6			3	Nanoparticles
7		8 Hours	1	Nanoparticles
8			2	Nanotubes, Nanospheres
9			3	Nanoparticles
10	1000°C	4 Hours	1	Nanoparticles
11			2	Nanospheres, Nano-triangular, Nanorods
12			3	Nanospheres, Nano-triangular
13		6 Hours	1	Nanoparticles
14			2	Nanoparticles, Nanospheres
15			3	Nanoparticles
16		8 Hours	1	Nanoparticles, Nanorods
17			2	Nanorods, Nanoparticles
18			3	Nanorods, Nanoparticles
19	1200°C	4 Hours	1	Nanoparticles
20			2	Nanospheres, Nanoparticles
21			3	Nanoparticles
22		6 Hours	1	Nanoparticles
23			2	Nanocrystal, Nano-triangular
24			3	Nanoparticles
25		8 Hours	1	Nanoparticles
26			2	Nanospheres, Nanorods, Nanocrystal
27			3	Nanorods

Table 3.
The different shapes of nanomaterials with different temperatures and baking times.

The third variable that play the important role was the ratio of the source material. For that variable, the combination of two different materials was reported by Tibayan et al. [23, 29]. The study was used Ag/SnO_2 and HVPG as the method to synthesis nanomaterial. The variable study based on the ratio between Ag and SnO_2 where stoichiometric ratio mixtures of 0:5, 1:4, 2:3, 3:2, 4:1, and 5:0 were used. Since the different of material mixture were added, the results showed that different nano shape have been reported as seen in the **Figure 4**. The time for baking is 8 hours in the 800°C temperature condition.

Based on the previous work done by many researchers used HVPG to synthesis nanomaterials, it is shown that the HVPG method can ensure the pureness and the high quality of the nano shape due to the excellent sealing process and occurred in the vacuum condition.

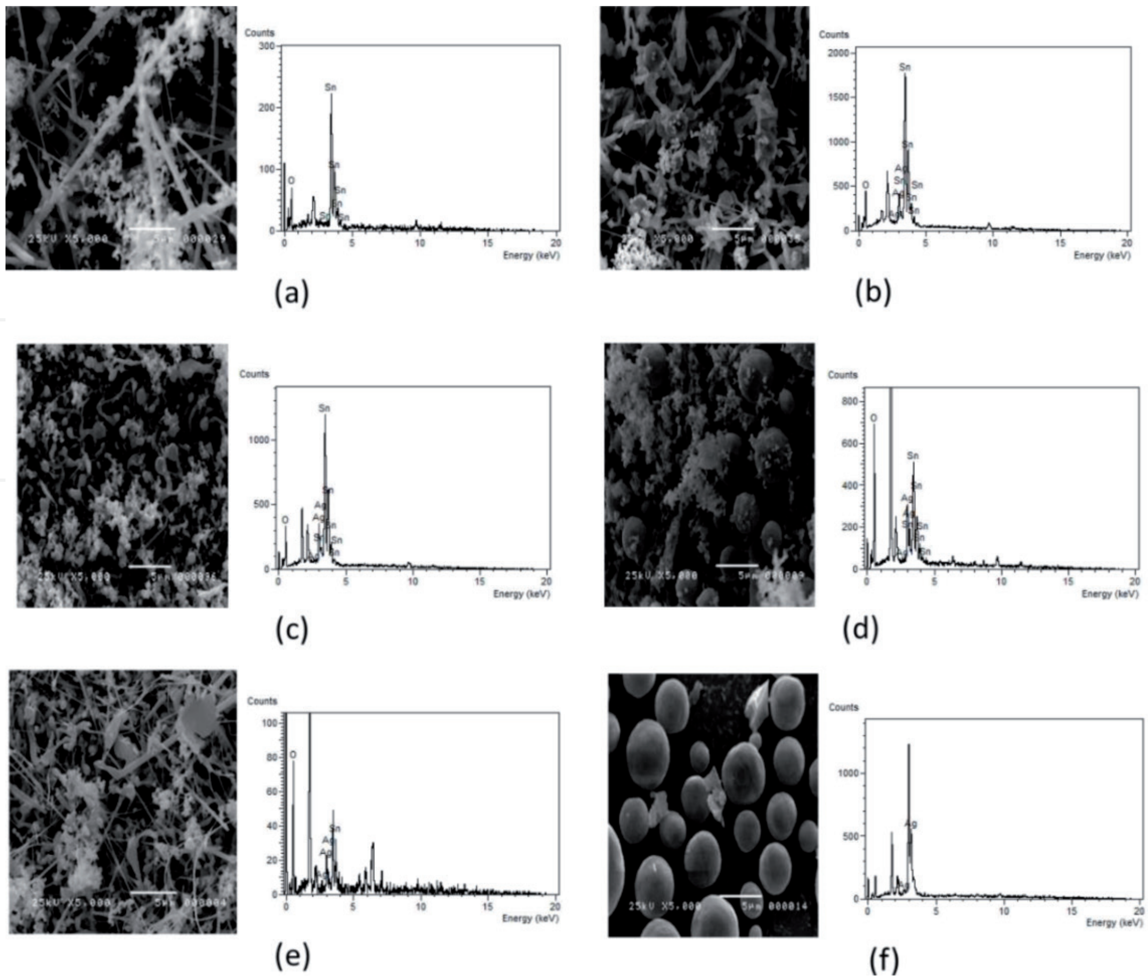

Figure 4.
Representative sample of Ag/SnO2 with different ratio. (Ag:SnO$_2$) (a) 0:5, (b) 1:4, (c) 2:3, (d) 3:2, (e) 4:1, (f) 5:0. Pictures were retrieved from [23].

4. Future trend

The pandemic that occurred in 2019 as known as COVID-19 has demonstrated the need for rapid, excellent, and robust technology that can prevent the virus and future diseases that may occur in the world. Researchers have been searching the new technology and they found that one of the best fits of the future technology that can be applied in almost all aspects of human life was founded in nanomaterials and nanotechnology [36, 37].

Nanotechnology become the most relevant solution for the problem of human life in the present day and the future due to the fact that nanotechnology has been proven to be applied in a different application. The various metal oxide was summarized by Shkodenko et al. [38] that useful to be applied as anti-bacterial technology. Pasquale et al. [37] were given an alternative perspective to disinfect the virus using a combination of TiO_2 nanomaterials in the photocatalytic process. It is shown that future applications can be nearly applied that photocatalysts for air, surface and water were available. Moreover, in medical based especially in dentistry, the graphene-based nanomaterial can be applied to eradicate microbial with good results [39]. Future manufacturing technology can potentially be combined with nanotechnology, as in additive manufacturing. Different nanomaterials, such as Carbon Nano Tubes, Carbon Nano Fibers, Graphene Oxide, Metal Nanoparticles, and Metal Oxide Nanoparticles, can be combined in the various polymer matrices [40]. It is shown that by adding nanomaterials, the properties of the matrices can be significantly improved. The field of automotive also become concerned by the scientist. Kotia et al. [41] reported that nanomaterials were used in automotive engine to improve the efficiency and performance of the machine. The future application in the field of optical sensing, biological imaging and photodynamic therapy was reported by Chen et al. [42]. The studies from many researchers were summarized and they reported that chemiluminescence resonance energy transfer platform based on nanomaterial successfully fabricated. The study related to drug delivery and toxicity have been evaluated by Jia et al. [43]. The nanomaterial was tested on the zebrafish to determine the effect of toxicity and biological related safety concerns. The in vivo toxicological profiles of different nanomaterials, including Ag nanoparticles (NPs), CuO NPs, silica NPs, polymeric NPs, quantum dots, nanoscale metal–organic frameworks, etc., that appear in zebrafish have been evaluated. Furthermore, mechanical testing related to strain sensing using graphene nanomaterial has been reported by Mehmood et al. [44]. Graphene nanomaterial has been chosen because its excellent properties in thermal, electrical and mechanical strength.

There are several aspect that related to nanomaterials. One of the most important aspects related to the synthesis of nanomaterials were about the environmental aspect where many syntheses processes were used catalysts or other materials that can harm the environment. To prevent the issue related to the environment, green synthesis was launched by many researchers as an alternative to producing nanomaterials. Different materials were introduced such as: biocompatible reagents, synthesis process by microorganisms, using plant mediated synthesis, improve the waster treatment system using nano filtering process, etc. [45]. It can be summarized that in the future, nanomaterials and nanotechnology still became the alternative and major material that used in various applications. In that point of view, HVPG technique that used to synthesis nanomaterial can be applied in further high scale synthesis process to fulfill the community needs related to nanomaterials [46].

5. Conclusion

The characteristics of synthesis nanomaterials using HVPG has been described and reported in the present study. The details aspect of the synthesis and the sample of the results of the synthesis nanomaterial also presented. It is shown that the HVPG can be used to synthesis various type of nanomaterials with the following advantages: excellent pureness ratio, free of external impurity during synthesis process, simple procedure and setup, environmentally friendly, and used recycle material (quartz tube) that previously used as the heating components. Since the application of nanomaterials can be found in very wide aspects, the synthesis process of nanomaterials using HVPG can be an alternative method. The future trend as shown in the present study ensure the sustainability of the synthesis nanomaterial without compromising with the environment and related to human healthy aspect.

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Conflict of interest

"The authors declare no conflict of interest."

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