# The Effects of Virgin and Recycled PA12 Powders in SLS Processes on Occupational Exposures

Amir Abdullah Muhamad Damanhuri, Azian Hariri, Sharin Ab Ghani, Mohd Syafiq Syazwan Mustafa, Safarudin Ghazali Herawan, and Nuur Azreen Paiman

***Abstract*—Particulate matter and ultrafine particles are emitted during the pre-processing and post-processing activities of selective laser sintering (SLS) processes, which is major concern to operators exposed to the powders. This study aims to determine the occupational exposure (in terms of the total particle concentration and respirable particulate concentration) during the pre-processing and post-processing activities of SLS processes using virgin and recycled polyamide 12 (PA12) powders. Personal air sampling was performed for each activity according to the NIOSH 0500 and NIOSH 0600 methods. Based on the results, both powders were uniform spheres with a particle size of 40–60 µm. The total particulate concentration was most significant during the following pre-processing activities: 1) pouring powder into the mixing machine and 2) transferring the powder to the SLS AM machine. The total particulate concentration and respirable particulate concentration were slightly higher for the virgin powder for these activities. In conclusion, the virgin and recycled PA12 powders were both inhalable and respirable, which poses serious health hazards to the SLS AM operators. Hence, it is essential for operators to use suitable personal protective equipment (including respirators) and the working practices need to be improved by automating some activities prevent manual powder handling.**

***Index Terms*—Occupational exposure, selective laser sintering, powder handling, polyamide 12.**

## I. Introduction

Additive manufacturing (AM) has been widely used in various industrial sectors such as aviation, biomedicine, automotive, and construction. With AM, manufacturers are able to realize complex three-dimensional (3D) objects based on the models drawn using a computer aided design (CAD) software. The model data stored in a Standard Tessellation Language (STL) file are used to print the objects. AM technology was introduced in 1986, with the development of stereolithography (SLA) by Charles and Hull [1]. In AM, the object, product, or prototype is created by joining materials layer by layer at a time [2], unlike subtractive manufacturing, where the material is cut (subtracted) from the block of material by grinding, rubbing, or drilling [3]. One of the advantages of AM is that it can significantly boost time savings by 50% in building prototypes. Another advantage of AM is its flexibility, where businesses can easily modify the prototypes and customize products to cater to different customers [4].

According to the American Society for Testing and Materials (ASTM) Committee F42 on Additive Manufacturing Technologies, there are seven types of AM technologies: 1) vat photopolymerization, 2) material jetting, 3) binder jetting, 4) material extrusion, 5) sheet lamination, 6) directed energy deposition, and 7) powder bed fusion (PBF). Selective laser sintering (SLS) AM machines is a form of PBF technology, which was introduced by Carl Deckard for his Master's thesis at the University of Texas in 1989 [5]. SLS AM processes can produce complex objects and prototypes with small and medium-sized geometries. In SLS processes, plastic, polymer, metal, ceramic, and/or composite powders are heated by a high-power directional heating source (laser) in the powder bed. The sintered powders will crystallize to create the object while the excess powders will be recycled and mixed with virgin powders in subsequent printing cycles [6], [7].

SLS processes consists of four stages, as shown in Fig. 1: 1) design (designing and modifying the 3D model in the CAD software and the model data are stored in the STL file), 2) pre-processing (weighing the powders, pouring the powders into the mixing machine, collecting the powders from the mixing machine, transferring the powders to the SLS AM machine), 3) processing (sintering the powders in the powder bed to create the object), and 4) post-processing (transferring the excess powders from the SLS AM machine, collecting the 3D objects from the powder bed, breaking the 3D objects from the powder cake, cleaning and polishing the 3D objects, cleaning the SLS process workplace, maintaining the SLS machine) [8].

Even though SLS processes offers numerous benefits such as reduction of material wastes and higher energy efficiency, there is growing concern on the impact of SLS processes on occupational safety [9], [10]. In SLS AM process, the pre-processing and post-processing activities are typically performed manually by the operators. Because the materials are in powder form (which are inhalable and respirable), they are hazardous to those involved in the SLS AM process and

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those within vicinity of the process.

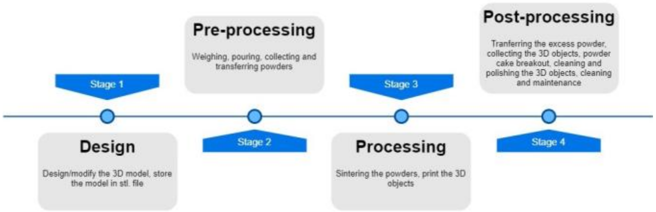



Fig. 1. Typical steps of SLS process.

Previous study [11] has shown that dry mechanical processes such as mixing, grinding, handling, and transferring powders will generate dust, which will significantly increase particulate matter emissions. Toxicological studies [12]–[14] confirmed that the operators of SLS AM machines are exposed to particulate matter and ultrafine particles from powder handling, where the particles enter their lungs and bloodstream through respiration. According to Väisänen *et al*. [15], PBF technologies increase the risk of health hazards, especially during handling of materials and objects. Graff *et al*. [16] evaluated the occupational exposure to virgin and recycled metal powders during SLS AM process. The results showed that the recycled metal powder was of smaller size compared with the virgin metal powder. In evaluating health hazards at the workplace, the occupational exposure needs to be assessed for as many single tasks as necessary. This enables preliminary and detailed investigation of the potential health hazards for all working conditions [17].

To date, only a few studies [15], [16] have been conducted to investigate the association between occupational exposure the use of virgin and recycled powders in SLS processes. Hence, the objective of this study was to assess the occupational exposure (in terms of the total particulate concentration and respirable particulate concentration) during the pre-processing and post-processing activities of the SLS process, all of which involve powder handling. Personal air sampling measurements were performed according to the NIOSH 0500 and NIOSH 0600 methods. Before the measurements, the powders were characterized to determine their surface morphology and particle size distribution. Polyamide 12 (otherwise known as nylon 12 or PA12) powder was chosen for the SLS process because it is a semi- crystalline polymer with favorable properties (high strength, high thermal stability, good fatigue resistance, and excellent surface resolution) [18]. The quantity for both virgin and recycled PA12 powders was fixed at 30 Kg for each experiment. Gravimetric analysis was performed for 8 h of SLS 3D printing. The findings of this study will provide insight on the effects of virgin and recycled PA12 powders on the total particulate concentration and respirable particulate concentration, which can be used to lay the foundation on improving working practices for operators involved in SLS processes to minimize occupational hazards.

## II. Methodology

### A. Experimental Setup

The PA12 powder (Farsoon FS33000PA) was purchased from a local supplier at a price of MYR 400/kg. The PA12 powder was supplied with the following specifications: 1) color: white, 2) bulk density: 0.4 g/cm$^3$, and 3) density: 0.95 g/cm$^3$ [19]. Herein, virgin powder refers to new (unused) powder whereas recycled powder refers to excess powder from the previous SLS AM processes. The quantity of the PA12 powder was fixed at 30 kg for each experiment. The occupational exposure during the SLS process was simulated and assessed at SLS Laboratory, Faculty of Mechanical and Manufacturing Engineering Technology, Universiti Teknikal Malaysia Melaka, Melaka, Malaysia. The SLS process workplace consisted of two 24-m$^2$ rooms, partitioned by a wall, as shown in Fig. 2. The pre-processing activities (weighing the powder, pouring the powder into the mixing machine, transferring the powder to the SLS machine, and printing the 3D parts) and post-processing activities (transferring the excess powder from the SLS machine / collecting the 3D parts from the powder bed and transferring the excess powder from the SLS machine, breaking the 3D parts from the powder cake / breaking the 3D parts from the powder cake, and cleaning and polishing the 3D parts, cleaning the SLS 3D printing process workplace (vacuuming, waste disposal, etc.)) were performed in Room 2, whereas the SLS processes was performed in Room 1. Each room was equipped with a split air conditioner, and the temperature and humidity were kept constant at 21 ℃ and 55%, respectively. In this industrial laboratory, SLS machines does not attached/nearby local exhaust ventilation and depended on general dilution ventilation based on 9 ACH. The pre-processing and post-processing activities are presented in Table I. The pouring height was fixed at 0.25 m [20]. The pre-processing and post-processing activities conform with those of other studies [15], [21], [22] which involved investigating the occupational exposure during powder handling in other industrial sectors. The occupational exposure (in terms of the total particulate concentration and respirable particulate concentration) was monitored during the pre-processing and post-processing activities [23]. Even though Activity D (printing the 3D parts) in Table I is technically not a pre-processing activity, it is included as a pre-processing activity for simplicity.

TABLE I: PRE-AND POST PROCESSING ACTIVITIES OF THE SLS PROCESSES AND THEIR CORRESPONDING TIME

| **Activities** | **Time of exposures (min)** |
|---|---|
| Pre-processing activities | |
| (a) Weighing the powder | 15 |
| (b) Pouring powder to the mixing machine | 30 |
| (c) Transferring the powder to SLS machine | 15 |
| (d) Printing 3D parts | 360 |
| Post processing activities | |
| (e) Collecting parts and excess powder from SLS machine, transferring | 15 |
| (f) Breaking 3D parts from powder cake | 30 |
| (g) Cleaning SLS workplaces | 15 |

### B. Characterization of Powders

Before the experiments, the virgin and recycled PA12 powders were characterized by scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and particle size analysis (PSA). SEM was performed to study the surface morphology of the powders. A scanning electron microscope (Model: TM 3000, Hitachi, Japan) with a magnification range of 5,000–500,000× and operating voltage range of 2.0–5.0 kV was used for this purpose. A thermogravimetric analyser (Model: STA 8000, Perkin Elmer, UK) was used to assess the thermal stability of a material based on its changes in mass as the material is heated at a constant heating rate. In this study, TGA was carried out by heating the sample from room temperature, 22 ℃ to 600 °C at a heating rate of 10 ℃/min. A particle size analyser (Zetasizer Nano ZSP, Malvern, UK) was used to determine the particle size distributions of the virgin and recycled PA12 powders. For this analysis, both types of powders were collected and the particle size distributions were measured before the SLS process [24]–[26].

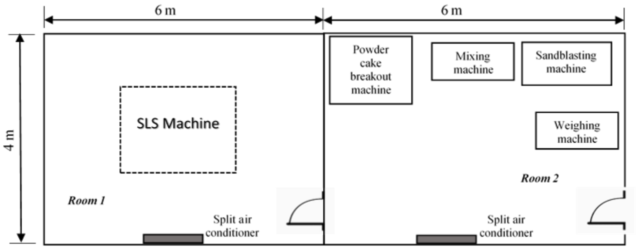


Fig. 2. Layout of SLS 3D printing workplace.

### C. Gravimetric Analysis of Exposures

Gravimetric analysis is a sampling technique used to evaluate occupational exposure. The air sample was collected in the operator’s breathing zone, which was within a 30-cm radius of the nose and mouth area. For gravimetric analysis, the operator is required to wear the sampling tools (filter, filter cassette, cyclone (where applicable), personal sampling pump, and flexible connecting tubes), which are fastened at the designated location on the operator’s body. The samples were acquired according to two air sampling methods by the National Institute of Occupational Safety and Health (NIOSH): (1) NIOSH 0500 and (2) NIOSH 0600. For the NIOSH 0500 method, the particle mass concentration was measured using a polycarbonate filter (diameter: 37 mm, pore size: 5 µm, SKC Inc., USA) in an open-face filter cassette. The flow rate of the personal sampling pump was set at 1.0 L/min. For the NIOSH 0600 method, the particle mass concentration was measured by using a polyvinylchloride filter (diameter: 37 mm, pore size: 5 µm, SKC Inc., USA) and 10-mm nylon cyclone. The flow rate of the personal sampling pump was set at 1.7 L/min. After sampling, the filter cassette was sealed with a parafilm and stored in a dry cabinet (temperature: 20±1 ℃, relative humidity: 50±5%) [27], [28]. The air sampling results represent the air inhaled by the operator.

A microbalance (Model: Quintixx65-1S, Sartorius, Germany) with a readability (resolution) of 1 µg was used for the gravimetric analysis. The gravimetric analysis was performed according to the NIOSH 0500 and NIOSH 0600 methods. The total particulate concentration (NIOSH 0500) and respirable particulate concentration (NIOSH 0600), both of which are the parameters used to assess the occupational exposure during the pre-processing and post-processing activities of the SLS processes, were determined using the following equation:

$$C = \frac{(W_2 - W_1) - (B_2 - B_1)}{V} \times 10^3 \qquad (1)$$

where $C$ is the total particulate concentration (NIOSH 0500) and respirable particulate concentration (NIOSH 0600) in the sampled air volume in milligrams per cubic meters (mg/m$^3$), $W_1$ is the tare weight of the filter before sampling in milligrams (mg), $W_2$ is the weight of the sample-containing filter after sampling in milligrams (mg), $B_1$ is the mean tare weight of the blank filters in milligrams (mg), $B_2$ is the mean weight of the blank filters after sampling in milligrams (mg), and $V$ is the sampled air volume at the nominal flow rate (1.0 L/min (NIOSH 0500), 1.7 L/min (NIOSH 0600)).

The time-weighted average (TWA) concentration for the pre-processing and post-processing activities of the SLS 3D printing process was determined using the following equation:

$$TWA = \frac{C_1T_1 + C_2T_2 + \dots C_nT_n}{TTE} \qquad (2)$$

where $TWA$ is the TWA concentration that the operator is exposed to over a specific period during the pre-processing and post-processing activities of the SLS processes in milligrams per cubic meters (mg/m$^3$), $C$ is the total particulate concentration or respirable particulate concentration in milligrams per cubic meters (mg/m$^3$) determined from Equation 1, $T$ is the time of occupational exposure in minutes per hour (min/h), and $TTE$ is the total time of occupational exposure [29], [30].

## III. Result and Discussion

### A. Morphology and Particle Size Distribution

Fig. 3(a) and (b) show the morphologies of the virgin and recycled PA12 powders, respectively, where the images were taken at a magnification of 2000×. The particles in both types of powders were uniform spheres. According to Chen *et al.* [31], spherical particles are conducive to powder flowability, which facilitates spreading in the powder bed of the SLS machine. There were large voids present in the recycled PA12 powder, which was expected because the powder was previously sintered at a high temperature (200-220 °C). Even though some voids were present in the virgin PA12 powder (Fig. 3(a)), these voids were not as pronounced as those in the recycled PA12 powder (Fig. 3(b)). The origin of these voids is not really clear, but it may be caused by absorbed moisture from the environment.

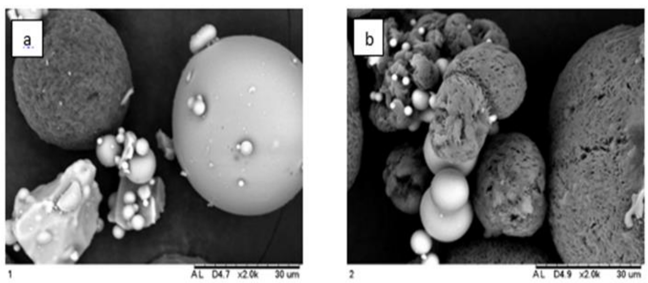


Fig. 3. Scanning electron micrographs of the (a) virgin PA12 powder and (b) recycled PA12 powder at magnification of 2000×.

Fig. 4 shows the particle size distributions of the virgin and recycled PA12 powders obtained from the PSA. In general, most of the particles for the virgin and recycled powders had a size of 40–60 µm However, the recycled powder had a higher volume density of particles at a particle size of 60 µm (13%) compared with the virgin powder. The virgin powder had a more uniform size distribution and had the highest volume density at a particle size of 50 µm (~10%). The results conform well with those of Dadbakhsh *et al*. [32], who found that there were no significant differences in the size and shape between the virgin and recycled PA12 powders. Based on the particle size distributions, the particles for both virgin and recycled PA12 powders are fine particulate matter, which identify as potential occupational exposure to the SLS operators especially during powder handling [33].

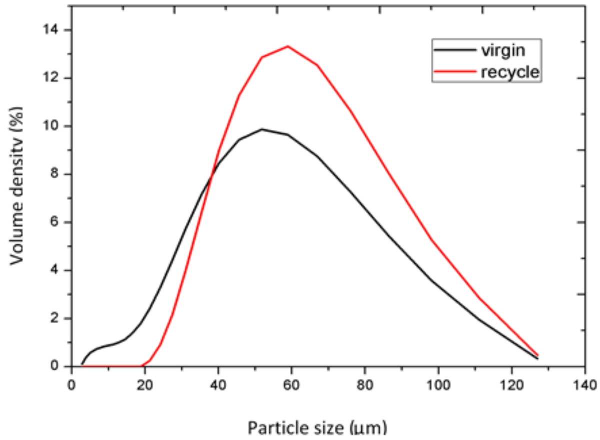
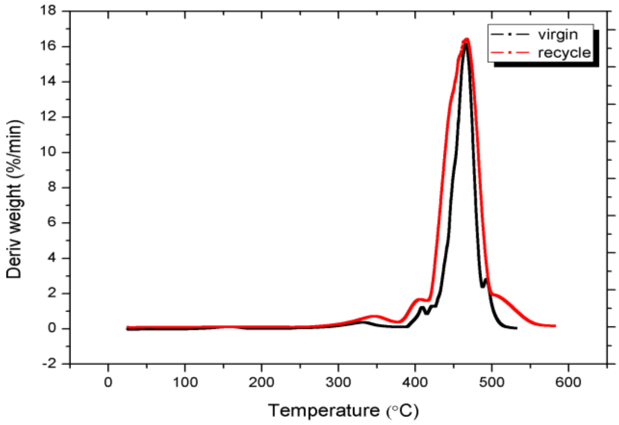
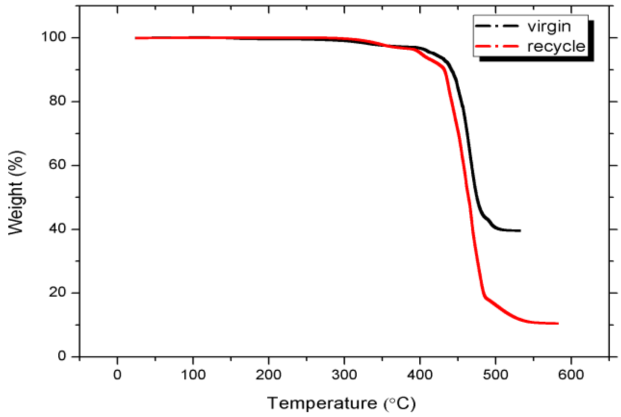


Fig. 4. Particle size distribution of virgin and recycled PA12 powders obtained from PSA.

Fig. 5(a) and (b) show the derivative thermogravimetric (DTG) and thermogravimetric analysis (TGA) curves of the virgin and recycled PA12 powders, respectively. Based on Fig. 7(a), it can be deduced that there were no significant changes in the PA12 powder after it was sintered at the normal sintering temperature of 220 ℃ since the DTG curves for the virgin and recycled PA12 powders were nearly coincident. As shown in Fig. 5(b), there were a few stages of weight loss: (1) initial weight loss at ~300 ℃, indicating that there was a low level of water absorption (~10%), (2) second weight loss of 10% at 300–400 ℃, (3) third weight loss at 400–480 ℃, which was most pronounced during the thermal decomposition process, and (4) final mass loss at 550 ℃. The TGA curves indicate that the PA12 powder spreads continuously during the SLS process at ~220 ℃ [28], [34], [35].



(a)



(b)

Fig. 5. (a) DTG curves and (b) TGA curves of the virgin and recycled PA12 powders.

### B. Gravimetric Analysis of Exposures

The total particulate concentration (NIOSH 0500) during the pre-processing and post-processing activities of the SLS process were determined for both virgin and recycled PA12 powders, and the results are presented in Table II. For the pre-processing activities, the total particulate concentration increased from Activity A (weighing the powder) to Activity C (transferring the powder to the SLS machine). In contrast, the total particulate concentration was the lowest for Activity D (printing the 3D parts), where the values were half of those for Activity B and C. The total particulate concentrations for Activity D were ±0.13 mg/m$^3$ and ±0.11 mg/m$^3$ for the virgin and recycled powders, respectively. In addition, the total particulate concentration was slightly higher for the virgin powder compared with that for the recycle powder for all pre-processing activities except for Activity D. The total particulate concentration was higher during Activities B and C (pouring the powder into the mixing machine and transferring the powder to the SLS machine), as expected. The results conform with those of Zisook *et al*.[14] and Vaisanen *et al*. [15], which present that highest dust concentration appear during period where operators manually handle the powder. . The pouring activity was found to increase the dust concentration during the mixing process.

For the post-processing activities, the total particulate concentration was highest for Activity F (breaking the 3D parts from the powder cake / breaking the 3D parts from the powder cake, and cleaning and polishing the 3D parts), with a value of ±0.268 mg/m$^3$. This is indeed expected because the operators will break the powder cake manually to remove the printed 3D parts during this activity, which will release significant amounts of dust. The total particulate concentration was slightly lower (±0.245 mg/m$^3$) for Activity G compared with Activity F because the operators will clean the SLS process workplace including vacuuming the rooms and disposing wastes. It shall be noted that for the post-processing activities, the PA12 powder is neither virgin nor recycled because the powder has already been sintered during the SLS process.

Likewise, the respirable particulate concentration (NIOSH 0600) during the pre-processing and post-processing activities of the SLS process were determined for both virgin and recycled PA12 powders, and the results are presented in Table III. In general, the respirable particulate concentration was higher for the pre-processing activities (Activities A–D) compared with the post-processing activities (Activities E–G). The respirable particulate concentration was most pronounced during the following pre-processing activities: (1) Activity B (pouring powder into the mixing machine) and (2) Activity C (transferring powder to the SLS machine). This indicates that the operator(s) are at a higher risk of health hazards from respirable particulates during the pre-processing activities. Based on the results for the total particulate concentration, the respirable particulate concentration was also higher for the virgin powder compared with the recycled powder for all pre-processing activities. For the post-processing activities, the respirable particulate concentrations were ±0.157 mg/m$^3$, ±0.149 mg/m$^3$, and ±0.168 mg/m$^3$ for Activities E, F, and G, respectively. The values were lower than those for the total particle concentration.

TABLE II: Mean Total Exposures for Pre and Post Processing Activities of the SLS Obtained According to NIOSH 0500 Air Sampling Method

| Activities | Mean total particulate concentration ±5% (mg/m$^3$) | Mean total particulate concentration ±5% (mg/m$^3$) |
|---|---|---|
| Pre-processing | Virgin | Recycled |
| (a) | 0.151 | 0.120 |
| (b) | 0.351 | 0.295 |
| (c) | 0.340 | 0.322 |
| (d) | 0.130 | 0.110 |
| Post processing | | |
| (e) | 0.173 | |
| (f) | 0.268 | |
| (g) | 0.245 | |

TABLE III: Mean Total Exposures for Pre and Post Processing Activities of the SLS Obtained According to NIOSH 0600 Air Sampling Method

| Activities | Mean total particulate concentration ±5% (mg/m$^3$) | Mean total particulate concentration ±5% (mg/m$^3$) |
|---|---|---|
| Pre-processing | Virgin | Recycled |
| (a) | 0.180 | 0.142 |
| (b) | 0.247 | 0.239 |
| (c) | 0.288 | 0.229 |
| (d) | 0.115 | 0.104 |
| Post processing | | |
| (e) | 0.157 | |
| (f) | 0.149 | |
| (g) | 0.168 | |

The TWA total particulate concentration and TWA respirable particulate concentration for both virgin and recycled PA12 powders are summarized in Table IV. The following observations were made. First, the TWA total particulate concentration for the virgin powder was slightly higher (0.1646 mg/m$^3$) than that for the recycled powder (0.1391 mg/m$^3$). Second, the TWA respirable particulate concentration for the virgin powder was also slightly higher (0.1357 mg/m$^3$) than that for the recycled powder (0.124 mg/m$^3$). Third, the TWA respirable particulate concentration was lower than the TWA total particulate concentration for both virgin and recycled powders. The results indicate that respirable particulates constitute a major portion of the particulates when recycled PA12 powder is used for SLS processes, since the TWA total particulate concentration does not differ significantly from the TWA respirable particulate concentration. Based on the results, it can be deduced that the operators are at risk of being exposed to particulate matter during powder handling. Occupational Safety and Health Administration (OSHA) have set permissible exposure limit (PEL) for total dust (15 mg/m$^3$) and respirable (5 mg/m$^3$) for 8-hour TWA limit for workplace exposures. In this study, despite the TWA concentration for all samplings are below PEL, safe handling and personal protective equipment are still needed.

TABLE IV: Comparison of the TWA Concentrations between the Virgin and Recycled PA12 Powders

| TWA (mg/m$^3$) | Virgin | Recycled |
|---|---|---|
| NIOSH 0500 | 0.1646 | 0.1391 |
| NIOSH 0600 | 0.1357 | 0.1240 |

## IV. Conclusion

In this study, the occupational exposure during the pre-processing and post-processing activities of SLS process (all of which involve powder handling) were investigated for virgin and recycled PA12 powders. Characterization was carried out on both powders before the indoor air sampling experiments. The SEM images revealed that the PA12 powder particles were uniform spheres; however, the presence of voids was more apparent for the recycled powder. The PSA results showed that the diameter of most of the PA12 powder particles was within a range of 40–60 µm, indicating that the operators are exposed to both inhalable and respirable particulate matter during powder handling. The TGA results revealed that there was no significant thermal decomposition of the virgin and recycled powders at the normal sintering temperature of 220 ℃.

Personal air sampling experiments were performed according to the NIOSH 0500 and NIOSH 0600 methods. Based on the gravimetric analysis results, the total particulate concentration and respirable particulate concentration were most significant during the following pre-processing activities: 1) Activity B (pouring powder into the mixing machine) and 2) Activity C (transferring powder to the SLS machine). For these activities, the total particulate concentration and respirable particulate concentration were higher for the recycled powder compared with those for the virgin powder. Both the total particulate concentration and respirable particulate concentration were the lowest during Activity D (printing the 3D parts). The TWA total particulate concentration and TWA particulate concentration were also determined. The results indicate that respirable particulates constitute a major portion of the particulate matter when recycled PA12 powder is used for SLSprocesses, since the TWA total particulate concentration does not differ significantly from the TWA respirable particulate concentration. The results of this study can be used to lay the foundation to promote safer working practices during powder handling of the SLS process, including the use of proper personal protective equipment.

## CONFLICT OF INTEREST

The authors declare no conflict interest.

## AUTHOR CONTRIBUTIONS

Amir Abdullah Muhamad Damanhuri conducted data collection and wrote the first draft paper. Mohd Syafiq Syazwan Mustafa, and Nuur Azreen Paiman help in preparing samples, setup instruments and data collection. Meanwhile, Dr. Azian Hariri, Dr. Sharin Ab. Ghani and Dr. Safarudin Ghazali Herawan help in content and paper reviewed.

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