Mixing and Reuse of Polymer Laser Sintering Powders to Ensure Homogeneity -A Review

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Mixing and Reuse of Polymer Laser Sintering Powders to Ensure Homogeneity – A Review

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Abstract

Selective laser sintering is one of the most common additive manufacturing technologies for polymeric materials. However, the technique is limited due to the high cost of the few available feedstock materials. Besides, laser sintering is a high-temperature process, which subjects polymeric materials to thermal stresses, resulting in the degeneration of powder. Besides, only a small portion of the powder fed into the built chamber is used in the printing of components. Hence, the need to recycle the used polymeric powder. This is achieved by mixing used and virgin powder in the ratios stipulated by the manufacturers of the powders. This review investigates the types of powder, types of mixing mechanism, parameters that affect efficient mixing, the homogeneity of mixed powder, mechanisms of segregation, and selection of the most appropriate mixers in the context of selective laser sintering of polymeric materials.

Keywords: selective laser sintering, polymers, degeneration of powder, mixing, used and virgin powder, homogeneity

I. INTRODUCTION

Additive manufacturing (AM) has redefined the field of production because it has enabled the development of geometrically intricate parts, which could not be achieved via conventional techniques. Besides, AM processes can be applied to a wide range of engineering materials such as metals, ceramics, and polymers [1]. Selective laser sintering (SLS) is one of the commonly utilized techniques in the AM of polymers. The technology is used to develop 3D parts through layer by layer fusion of powder material using a laser beam [2]. The process commences by pre-heating polymeric materials to a temperature just below their melting point to ensure that a beam of less energy density can be used to produce parts because high energy density leads to the deterioration of the properties of polymer powder material [3]. After this, a laser beam is used to melt and fuse the particles of powder selectively. During the process of sintering, high temperatures between the melting and crystallization point of the material should be maintained in the build chamber to prevent warping of the built parts and ensure good surface finish [2]. Therefore, the powder material is subjected to high temperatures in the build chamber for prolonged periods, which leads to degradation of the powder. Consequently, aged polymeric powders, such as polyamide 12 (PA 12) material, have less flowability, which in turn affects the laser sintering process and quality of the finished product in terms of mechanical properties, surface finish, and dimensional accuracy [3]. For this reason, the used powder is recycled by mixing it with virgin powder to ensure the suitability of the mixture for reuse. Research work by Duddleston [25], illustrated that increasing virgin powder returns the melting temperature of the used powder, to almost that of virgin powder (the green horizontal line), as demonstrated by Figure 1, which shows the melting point of differently mixed PA12 materials (0%, 20%, 40%, and 60%, fresh material) sintered for different durations of 12, 24, 48, and 96 hours.

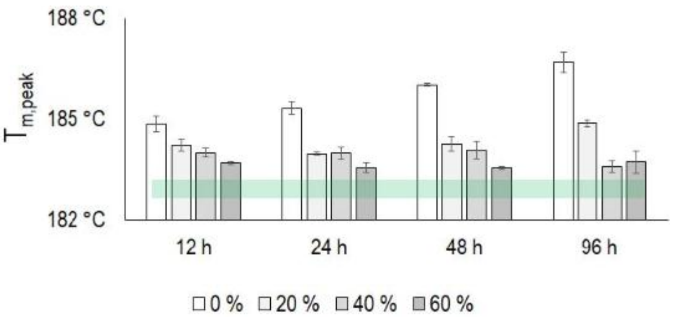

Fig. 1. Effect of adding different percentages of virgin material to used PA12 [25].

The term, used powder, refers to cake, overflow, and supply powder that is mixed with the first two. The supply and overflow powders are less damaged than the cake powder, which supports the printed parts. Therefore, sufficient mixing is required to ensure homogeneity and uniformity of recycled supply powder.

There is a limited variety of polymers with acceptable properties for use in SLS due to stringent material and sophisticated processing requirements [1]. Furthermore, in SLS, 90% of the polymers used are polyamides or polyamide blends. However, other materials, such as polypropylene, thermoplastic polyurethane (TPU), polyether block amide (PEBA), and polyether ether ketone (PEEK), are being introduced into this field [1]. Hence, the cost of the available commercial laser sintering polymeric powders is relatively high. For example, the cost of one kilogram of PA 12 (PA 2200) from EOS GmbH is about $88 [4]. Therefore, there is a need to recycle the used polymer powders to reduce the cost of production.

According to the article by Yamauchi, Kigure, & Niino [5], the amount of cake powder is usually about 20% of the supplied powder. This ratio is referred to as the recyclability of the powder [5]. There is a need to recycle the un-sintered power to reduce wastage. Therefore, after sintering, the cake powder is sieved, stored, and used for the next print cycle. However, the cake powder cannot be used on its own due to material degradation [5] and is mixed with the un-used powder to restore its flowability.

A lot of research has been conducted to investigate the effect of laser operating conditions and the properties of powder on the quality of the final product [1, 2, 3, 5, 7, 8, 9, 10, 11, 12, 13]. However, little effort has been conducted to investigate the effect of mixing of the used and the virgin powders in terms of homogeneity of the ready powder. Most of the available research has, in many cases, been conducted to analyse blending of pharmaceutical products. Thus, there is limited study on the homogeneity aspect of mixing of laser sintering used and virgin polymeric powders in SLS. It is important to ensure that the mixing process is effective in ensuring homogeneity of the mixed powder. However, the process of mixing powders poses several challenges due to the unpredictable and irregular properties of powders, such as varying particle size, particle shape, size distribution, particle density, and cohesivity [14]. These factors influence the homogeneity of mixed powders.

As mentioned, few studies have focused on the homogeneity of the mixture of polymeric powders used in the SLS process**.** To this end, the ensuing discussion provides a brief overview of the type of powders, types of mixing mechanism, parameters that affect efficient mixing, mixing in rotating cylinders, sampling of mixed powder, assessment of the homogeneity of powder, mechanisms of segregation and selection of the most appropriate mixers.

II. RECYCLING OF POLYMERIC MATERIALS USED IN SELECTIVE LASER SINTERING

The recycling process involves mixing of the used powder with virgin powder in proportions that ensure the mixed powder has an acceptable melt flow rate (MFR) [6]. The recommended virgin versus used powder proportions are as illustrated by Figure 2. The mixing process and ratios are important because they determine the quality of mixed powder that is ready to use, which in turn dictates the quality of products printed in terms of strength, surface finish, and dimensional accuracy [7]. Table 1 summarizes the recommended refresh rates (ratios of cake to virgin powder) for the most commonly used SLS polymers, while Figure 2 shows the mixing paradigm used by many SLS polymer manufacturers.

Table 1. Recommended Refresh Rates [6]

Manufacturer/ material name	Refresh rate fresh powder (%)	Additional recommendations
EOS GmbH		
PA2200 fine polyamide	30 to 50	Scrap the powder if there is severe "orange peel" texture
PA3200 GF polyamide	50 to 70	
3D Systems Corporation		
DuraForm (polyamide)	30 + (30 overflow)	Scrap the powder if there is severe "orange peel" texture, normally every 8-10 builds
GF DuraForm	50 + (overflow)	

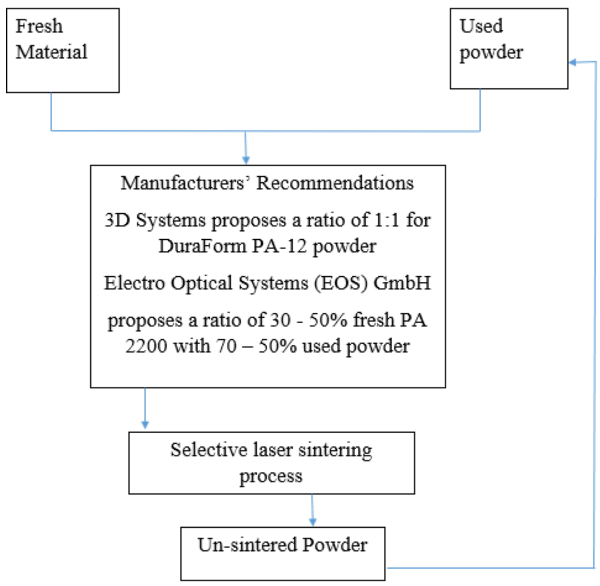

Fig. 2. Typical recycling process for SLS polymeric powders [6].

According to an article by Dotchev & Yusoff [6], when PA2200 is recycled several times without or with small quantities of new material, the produced parts have a rough surface finish. This phenomenon is referred to as orange peel. Another study by Gornet [15] illustrated that the MFR of PA 2200 reduced from 50 to 20 g/10 min when recycled without blending it with virgin powder for 7 builds. The study further found that the tensile strength of the manufactured parts increased with reuse cycles. However, there was high part shrinkage, which affected the surface finish and dimensional accuracy. Dotchev & Yusoff [6], confirmed that the current powder recycling practices do not consider the variation of the properties of the used powders. Hence, the need to further

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investigate mixing of polymeric powders used in SLS.

Figure 3 shows a minimum recommended MFR for PA 12 of about 25 g/10min. Furthermore, a power grade with MFR below 18 g/10min is considered as aged material and should be discarded [6]. It can also be deduced that the MFR value of a mixture of polymeric powder is dependent on the degree of degradation of the material, as well as the ratio between fresh and aged powder. A high percentage of used powder increases the MFR of a mixture, which reduces the flowability of the powder, and in turn impedes laser sintering of the materials. Therefore, there is a need to investigate the dynamics surrounding mixing of polymeric powders for SLS processing.

PA2200 powder grades	MFR (g/10 min)
A (fresh powder)	More than 50
B1	45-49
B2	40-44
B3	35-39
B4	30-34
B5	25-29
B6	18-24
C (old powder)	Less than 18

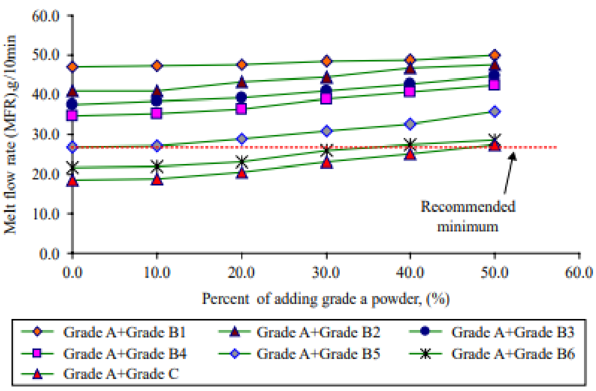

Fig. 3. Effect of adding different PA 12 grades on MFR [5].

III. TYPE OF POWDERS

Powders can be categorized as either free-flowing, random, or ordered powders [14, 16]. Over the years, ordered powders have also been termed as interactive powders or adhesive powders [16]. The random powders have also been referred to as non-interactive powders or cohesive powders [16]. Figure 4 differentiates between ideally ordered and perfectly random mixtures.

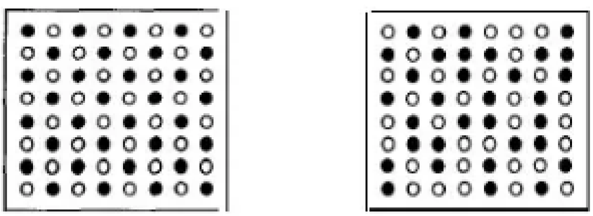

Fig. 4. The difference between ideally ordered (Left) and perfectly random mixtures (Right) [16].

Free-flowing powders move smoothly because of low inter-particulate forces. These powders have a problem of segregation after mixing. Therefore, they should be stored properly with minimal disturbance. Selective laser sintering polymeric powders should exhibit free-flowing behaviour, which encourages the spreading of the powders [10]. As a result, the printed parts are free from porosity and, in turn, possess excellent mechanical properties. Besides, free-flowing polymeric powder reduces the chances of the recoater blade dislodging printed parts from the building bed. Non-free flowing powders tend to clump together, leading to uneven deposition of powder on the powder bed, which leads to non-uniform powder bed density, and in turn, compromises the mechanical strength of printed parts [26].

Cohesive (random) powders cling to each other due to significant inter-particle forces. Cohesive powders are also formed due to moisture and electrostatic charges [14]. They do not exhibit the free-flow phenomenon. Besides, cohesive powders have low segregation tendencies and are difficult to mix properly. Hence, cohesive powders are not suitable for use in SLS.

Ordered (adhesive) powders have uniformly distributed particles and are formed when small particles adhere to large particles [14, 16]. Figure 6 shows an example of an ordered mixture. The concept of ordered powders is applied when developing polymer blends for SLS to ensure suitable properties.

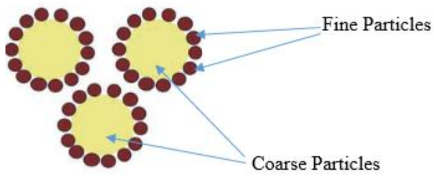

Fig. 5. An example of ordered mixture [17].

IV. TYPES OF MIXING MECHANISMS AND PARAMETERS THAT AFFECT EFFICIENT MIXING

Mixing occurs either through diffusion, shear, or convection [16, 18, 19]. In diffusive mixing, the powder particles are blended through rotation, where mixing is achieved through the motion of individual particles relative to one another [16]. In shear mixing, the particles are mixed in slip zones [18], while convective mixing involves mixing of powders by circulation patterns, where the particles are transferred from one location to another [20]. Furthermore, convective mixing is subject to the geometry of the mixing system and the presence of elements such as paddles, blades, and baffles [20]. Mixing of free-flowing powders occurs mainly through convective mixing [20]. Convective mechanisms are utilized for macro-mixing, while diffusive mechanisms apply mainly in micro-mixing [20]. In convective mixing, particles move relative to each other due to differences in velocity [21]. This leads to the formation of thin spaces, which are termed as failure zones [21]. Particles in the failure zone will move downwards if the weight of the particles is higher than the inter-particle cohesive force, while large particles move towards the failure zone [21]. The phenomenon leads to both mixing and segregation.

The process of mixing involves shuffling of both small and large particles to ensure that there is a uniform distribution of the particles in a given bulk material [14]. Therefore, the process involves a random distribution of particles for a given period, and after the desired mixing has been attained, the movement of the particles is halted by ensuring that the system is in a static equilibrium to avoid segregation [14]. Individual particles are redistributed within the bulk either by tumbling, shearing, scooping, kneading, or impaction.

The efficiency of mixing is affected by particle parameters, mixer parameters, and the segregation tendency of the particles. The particle parameters include particle size, particle shape, size distribution, particle density, and cohesivity [14]. Narrow powder particle distribution, as well as reduced absolute size and fewer irregularities, ensure homogeneity of a mixture of powder. Suitable SLS polymer powder particle sizes lie between 20 and 80 microns [1, 10]. Furthermore, the powder particles should possess an almost spherical shape to promote free-flowing behaviour, which encourages spreading of the powder during the SLS process [1, 10]. Differences in particle density also promote segregation during mixing, which reduces the homogeneity of powder mixture as heavier particles tend to be attracted to one another by gravitational force [22]. Finally, the inter-particle forces influence homogeneity if mixtures of cohesive powder pull towards each other, whereas adhesive particles repel from each other.

The mixer parameters that influence the homogeneity of a mixture of powders include speed, time, batch volume, and mixer angle [14]. According to a study by Hogg [20], an increase in speed increases the efficiency of powder mixing. The most effective mixing is obtained once the critical speed is attained for cylindrical mixers. In addition, longer times in the mixer ensure better mixing. However, the mixing time should be reasonable to avoid wasting time without gaining value in terms of the quality of mixing [19]. Moreover, Hogg [20] observed that increasing the volume of the powder lowers the effectiveness of mixing of the powder. Besides, the number and angle of blades of mixers relative to the position of the axis of rotation also affect the homogeneity of the mixed powder. Increasing the number and reducing the angle of blades encourages homogeneity because larger portions of the powder are subjected to the mixing action [19, 23]. Furthermore, if the axis of rotation is almost vertical, then the blades cannot lift the powder. Hence, poor mixing is achieved. The axis of rotation should be kept at about 15^0 from the horizontal to achieve efficient mixing of power [22]. Overall, speed, time, batch volume, and mixer angle affect the homogeneity of a mixture of powders.

During the mixing process, all particles in the system should have the same mobility. However, if there is a difference in their mobility, then separation of particles might occur. This phenomenon is referred to as powder segregation [20]. Mobility of particles is subject to gravity, resistance to motion, the environmental conditions, and the mixing device [20]. Convective mixing is unlikely to cause segregation because the process does not depend on the mobility of particles. However, the external force causing convective mixing might lead to the segregation of particles of powder [20]. Hence, mixing of powders might also contribute to non-uniform mixtures, which demands proper mixing parameters to be set.

Segregation of particles in a batch of powder depends on the size, shape, and density of the particles [16, 22]. During mixing, small particles tend to align together, while the coarser particles agglomerate together [16]. It has also been noted that more spherical particles tend to pull together in contrast to irregularly shaped particles [16, 22]. Therefore, particle segregation should be considered during mixing and storage of powders.

V. MIXING IN ROTATING CYLINDERS

Rotating cylinders are commonly used for mixing powders used in SLS, and the process can take place in either vertical, horizontal, or inclined positions. Study shows that for non-segregating powders, powders with the same size, shape, and density, vertical mixing is diffusive, whereas the horizontal mixing is a combination of convective and diffusive [20]. In the horizontal position, the powder batch is divided into two regions; the static region, where there is no mixing since the particles follow the motion of the cylinder, and the shear zone where the actual mixing takes place [20]. To ensure that the batch of powder behaves like a single entity, the Froude number of the mixture must be equal to 1 [20]. The Froude number describes the conditions in the shear zone and is calculated using the expression [20]:

$$Fr = R\omega^2/g \qquad (1)$$

where the symbol *Fr* stands for Froude number, *R* radius of the cylinder, ω angular velocity of the cylinder, and *g* acceleration due to gravitational.

The Froude number becomes 1 when critical speed is attained. Critical speed is calculated, as shown in Equation 2 [20].

$$\omega_c = \sqrt{g/R} \qquad (2)$$

where, ω_c stands for critical speed, g acceleration due to gravity, and *R* radius of the cylinder.

As mentioned earlier, with reference to vertical mixing, only diffusive mixing takes place. Notably, the diffusion coefficient is subject to the volume of the filled powder, particle size, and the rotational speed. According to Hogg [20], the diffusion coefficient decreases with an increase in the volume of powder being filled into the cylinder and an increase in particle size. The diffusion coefficient also increases with an increase in the rotational speed.

VI. SAMPLING OF MIXED POWDER AND ASSESSMENT OF THE HOMOGENEITY OF POWDER

The two sampling methods used are stratified and nested sampling [14]. The latter method involves selecting samples from a single point within a batch, whereas the stratified sampling method involves selection of samples from different locations in a given batch of powder [14]. Besides, samples should be obtained when the powder is in motion and within short time intervals to ensure a true representation of the powder [14]. Hence, stratified and nested are the two most commonly used sampling techniques in power technology.

It is essential to take five samples from different locations of the mixer [21]. For a horizontal mixer, samples should be obtained from each corner and one from the middle of the batch. In addition, one sample should be obtained at the discharge of the mixer [21]. Samples are mostly obtained using a thief probe, which is a metal rod with recessed cavities along its length. The rod is inserted into a batch of powder and samples collected in its cavities. The effectiveness of the technique is depended on the operator's experience and other factors such as the insertion angle, penetration rate, and twisting effect [21]. Overall, the sampling process is crucial since it determines the accuracy of the samples collected.

The homogeneity of a batch of powder can be determined by taking several samples at random and then obtaining the variance of the composition of each sample [20]. Different technologies can be used to determine the effectiveness of a mixing process. These technologies include; the use of a standard granulometric analyzer, which is used to determine the average size of particles for a given sample [24]. A laser particle analyzer is also used to determine the distribution of particles for each sample. Scanning electron microscopy can be used to determine the distribution of particles in each sample. The results are analysed statistically to determine the variation. A homogenous powder batch should have zero standard deviation for all the samples considered [24].

VII. SELECTION OF A MIXER FOR SELECTIVE LASER SINTERING, POLYMERIC POWDERS

The efficiency of mixing polymeric powders used in SLS is dependent of the mixer used. In this regard, a suitable mixer should possess the following characteristics [14]:

1. Be capable of producing a homogenous powder within a reasonable time frame and without damaging it.
2. Be dust tight.
3. Require low maintenance and energy.
4. Be easy to discharge and clean.
5. Be flexible for various batch sizes.

A flow chart for selection of a mixer is summarized in Figure 7.

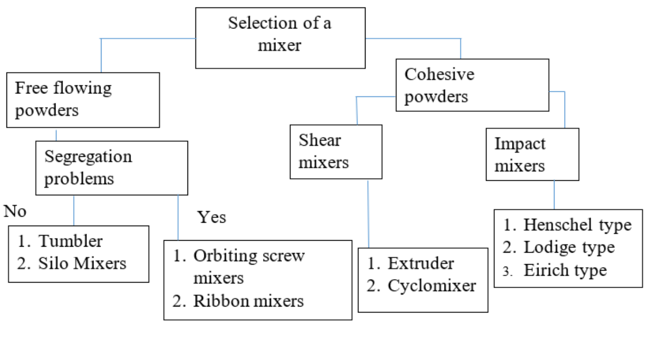

Fig. 6. Flow chart for the selection of a mixer [14]

Most of the available mixers have limitations such as loss of product, abrasion of the particles of powder, contamination of the powder, and inflexibility to batch sizes of powder [18]. Some low-shear mixers such as tumblers might not be effective to efficiently mix powders that are in an agglomerated state since they require to be broken-up first and then redistributed [20]. However, the tumbler mixers continue to be utilized in the industry because of their effective convective and diffusive mechanisms of blending, close quality control, and gentle mixing of powders [21]. Besides, the speed of most tumblers ranges between 5 and 25 revolutions per minute, with a fill level range of 50 to 75 % [21]. In addition, the shape of most tumblers is asymmetrical in shape to lower the effects of centripetal force (which is prevalent in circular shaped mixers) and hence ensure homogeneity. Tumbler mixers continue to be used for mixing purposes due to their advantages.

Selective laser sintering polymeric powders are mixed using tumbler type of mixers, in particular, concrete mixers. Most additive manufacturing centres, such as the Center for Rapid Prototyping and Manufacturing (CRPM) at the Central University of Technology (South Africa), utilize tilting drum mixers such as the one shown in Figure 8.

Fig. 7. A concrete mixer used at CRPM.

In these types of mixers, the mixture of powders is added to the drum, and then the drum is set to a suitable axis of rotation, preferably one that is inclined at 15^0 to the horizontal, as shown in Figure 9 [23]. The powder is then mixed by rotating the mixer for an appropriate duration and then discharged, as shown in Figure 9. Poux et al. [16] proposed that the most suitable mixing time lies between 2 to 10 minutes.

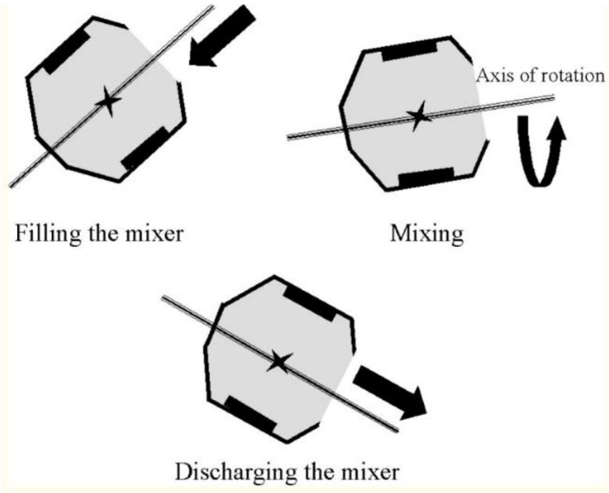

Fig. 8. Operating process of a tilting drum concrete mixer [23].

The drum concrete mixers are preferred for mixing SLS polymeric materials because they are easy to use and maintain. Moreover, they are relatively cheap to purchase and install. Furthermore, their speed is between 5 and 25 revolutions per minute, which reduced the chances of centripetal and centrifugal forces [23]. Besides, these types of mixers can handle relatively large amounts of powder.

VIII. CONCLUSION

- Selective laser sintering, which is widely used in the additive manufacturing of polymeric materials, results in the deterioration of the properties of polymeric powders since it is a high-thermal technology. Hence, the need to restore the properties of the infill powder and reduce the deleterious effects associated with SLS.
- The properties of aged powder are enhanced by mixing the used powder with fresh material.
- Suitable SLS polymeric powder should demonstrate free-flowing behaviour to promote spreading.
- The homogeneity of the used-fresh SLS polymeric powders is subject to the speed, time, batch volume, and mixer angle of concrete mixer equipment, which are commonly employed by many manufacturers.
- Homogeneity of mixed SLS polymeric powders is achieved:
  - When different batches are mixed for a duration of 2 to 10 minutes,
  - When the drum is inclined at about 15^0 to the horizontal,
  - When the fill level of the mixer is around 50 to 75%, and
  - When the rotating speed is between 5 and 25 revolutions per minute.

CONFLICT OF INTEREST

The researchers have no conflict of interest to disclose with regard to the current review.

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