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MODIFICATION OF POWDER MATERIAL FOR 3D PRINTING USING DRY GRANULATION

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Abstract

Additive technologies currently represent progressive technologies, both in the field of manufacturing itself and in materials engineering, where the main source is powder material. Powder material as a source material is characterized by the mechanical, physical and flow properties of fine powders. Targeted modification of these properties represents a research challenge in this area with the aim of creating a granulate with the required properties. This article compares selected mechanical, physical and flow properties of the original powder material used for 3D printing with the created granulate. The granulate was created by a process of high-pressure compaction between the rolls of a compactor and subsequent dry granulation on a patented flat-die granulator to create a granulate with a narrow particle size distribution. The result is an assessment of the improvement in flow properties for possible material recycling and reuse of the raw material for further processing using 3D printing.

Keywords:

Powder Material, Flow Properties, Dry Granulation

1 INTRODUCTION

1.1 Additive technologies

Additive technologies, also known as 3D printing, represent a revolutionary approach to manufacturing three-dimensional objects from digital models. Unlike traditional subtractive manufacturing processes, such as turning or milling, which remove material from a block, additive technologies add material layer by layer, creating complex structures with high precision. The advantages of additive technologies include speed and flexibility of production and reduced waste when manufacturing complex components. The usefulness also include ability to manufacture complex structures that could not be produced by traditional manufacturing processes. [Gibson 2021, Stucker 2016, Eckert 2022]

2 EXPERIMENTAL MATERIAL USED FOR MATERIAL RECOVERY

Additive technologies can utilize a wide range of materials, including plastics, metals, ceramics, and composites. The choice of material depends on the desired properties of the

object, such as strength, temperature resistance, and chemical resistance.

The experimental material used for the production of granulate for material recovery was PA2200 (PA12) powder, which is used in additive manufacturing using Selective Laser Sintering (SLS) technology. Selective Laser Sintering (SLS) is a 3D printing technology that uses a high-powered laser to selectively sinter (fuse) powder particles layer by layer, creating a three-dimensional object based on a digital model. SLS technology can also utilize wider range of nylon-based materials such as PA1101 (PA11), PA 3200 GF, PA 2210 FR, Alumide – PA12 and some others which are well studied.

PA2200 is a polyamide material that is characterized by high strength, temperature resistance, and chemical resistance. PA2200 is suitable for the production of functional prototypes, tools, and end-use products in various industries, such as automotive, aerospace, and medical. The properties of PA2200 material are listed in Table 1. Also in article [Kozior 2020] is stated deeper analysis of the PA2200 powder and its properties with rheological focus. [Krupova 2024, Zarybnicka 2022, EPFL 2024, VEXMA 2024, Malashin 2024]

3 GRANULATION OF POWDER MATERIAL OF PA2200

3.1 High pressure compaction

The used powder material was compacted on a powder material compactor at the Institute of Process Engineering, Faculty of Mechanical Engineering, Slovak University of Technology in Bratislava. The compactor was designed and constructed scientific research activities with the possibility of setting parameters such as the frequency of rotation of the trains, the frequency of rotation of the screw for forced filling. The output is the compacting pressure between the rolls, which allows the strength of the granulate to be adjusted. [Peciar 2017, Salman 2007, Gupta 2005, McAuliffe 2015, Acevedo 2012, Wagner 2013, Cohn 1966]

3.2 Dry granulation

Dry granulation in this case represented granulation on a patented device Granulator of particle material with flat matrix, which was designed and constructed at the Institute of Process Engineering, Faculty of Mechanical Engineering, Slovak University of Technology in Bratislava. The granulator pushes a compacted material from the high-pressure compaction process through a perforated flat matrix using inclined blades. This process generates a very low percentage of dust and therefore the efficiency of the equipment is very high. Two matrix with different mesh sizes were used in the granulation process. [Salman 2007, Peciar 2018, Palugan 2022, Arndt 2018, Sun 2016]

3.3 Sieving process

The aim of the sieving process of the produced granulate was to create a narrow distribution of the individual fractions, with the target of creating a fraction with $D_{50}(1) = 1.0$ mm and $D_{50}(2) = 1.8$ mm. The Retsch AS200 batch sieving device was used for the sieving process.

The original PA2200 powder material and the two created granulates are shown in Figure 1.



Fig. 1: PA2200 particulate material.

- a) original powder, b) granulate no. 1 - $D_{50}(1) = 1.0$ mm;
c) granulate no. 2 - $D_{50}(2) = 1.8$ mm.

4 COMPARISON OF SELECTED PROPERTIES OF PA2200 BEFORE AND AFTER GRANULATION

A comparison of the material before and after the granulation process was performed by analyzing the particle size distribution and flow properties (angle of internal friction, flow function, Hausner ratio) of the powder material and the granulates.

4.1 Particle size distribution

The PartAn3D device from Microtrac was used to analyze the particle size distribution. The instrument is used for dynamic particle size measurement in the range of 15 μ m to 35 mm. The comparison of the particle size distribution of the original PA2200 powder material and the two created granulates is shown in Figure 2. The original powder has a D_{50} parameter of 0.06 mm, granulate no.1 has a D_{50} parameter of 1.0 mm, and granulate no.2 has a D_{50} parameter of 1.8 mm. [MICROTRAC 2015]

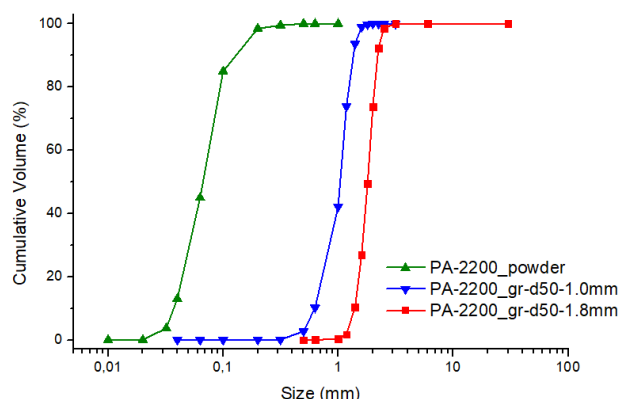


Fig. 2: Particle size distribution of PA2200 powder and two PA2200 granulates

4.2 Angle of internal friction

The Freeman FT4 from Freeman Technologies was used to analyse the internal friction angle using a 50 mm diameter chamber. A comparison of the fractional composition results of the original PA2200 material powder and the two granules produced is shown in Figure 3. [FREEMAN 2020]



a)



b)

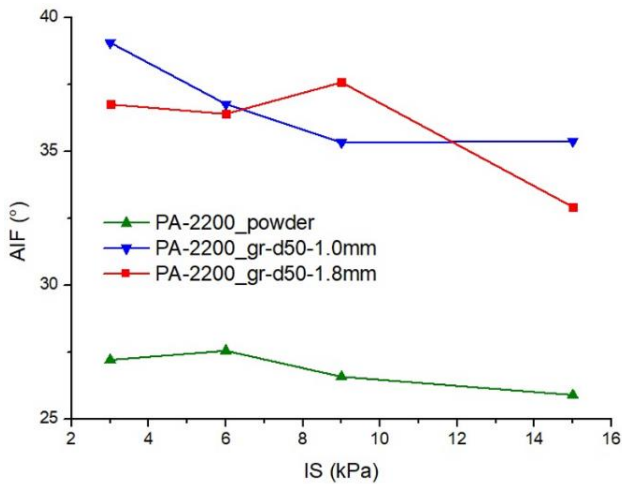


Fig. 3: Angle of internal friction (AIF) of PA2200 powder and two PA2200 granulates

4.3 Flow function

Shear testing using the Freeman FT4 instrument enabled the construction of flow functions for the experimental material. These flow functions provide a characterization of the powder material's flow behavior. The methodology employed aligns with the principles proposed by A.W. Jenike and comprehensively described by D. Schulze [FREEMAN 2020]. The measurement results are presented in Figure 4.

The flow function parameter ff_c is defined as

$$ff_c = \frac{MPS}{UYS} \quad (1)$$

where MPS (kPa) represents the major principal stress and UYS (kPa) denotes the unconfined yield strength measured during the shear test. The flow function parameter allows for the characterization of the flow properties of individual samples. The values ff_c within the range of 0 - 1 indicate non-flowing powder materials, 1 - 2 very cohesive powder materials, 2 - 4 cohesive materials, 4 - 10 easy-flowing materials; if the parameter $ff_c > 10$, the powder materials are free-flowing [Gupta 2005].

Classification of materials according to ff_c values is shown in Figure 5.

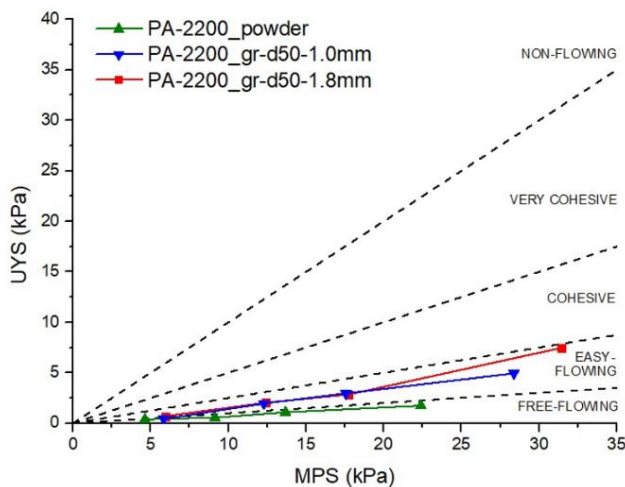


Fig. 5: The flow function parameter classification of PA2200 powder and two PA2200 granulates

4.4 Hausner ratio

Hausner ratio is a dimensionless quantity used to characterize the flow properties of powder materials. Hausner ratio is defined as:

$$HR = \frac{\rho_{tapped}}{\rho_{bulk}} \quad (2)$$

where ρ_{tapped} is the tapped density of the powder and ρ_{bulk} is the bulk density of the powder. The classification of particulate materials according to the Hausner ratio is shown in Table 2.

The Sotax Tapped Density Tester TD1 instrument was used to analyze the Hausner ratio, and the results for the measured powder and the created granulates are shown in Table 3.

Tab. 2: Powder flowability based on the Hausner ratio

Hausner ratio	Flow character
1.00 – 1.11	excellent
1.12 – 1.18	good
1.19 – 1.25	fair
1.26 – 1.34	passable
1.35 – 1.45	poor
1.46 – 1.59	very poor
more than 1.6	very, very poor

Tab. 3: Hausner ratio and flow character of PA2200 powder and two PA2200 granulates

Sample	Hausner ratio	Flow character
PA-2200_powder	1.136	good
PA-2200_gr-d50-1.0mm	1.154	good
PA-2200_gr-d50-1.8mm	1.144	good

5 DISCUSSION, RESULTS OF EXPERIMENTAL MEASUREMENTS

The basic flow properties of the created granulates, such as the angle of internal friction, flow function, and Hausner ratio, were evaluated by experimental measurements. The particle size distribution was also analyzed for the source powder and the created granulates.

Based on the experimental measurements, it was found that the PA2200 source powder is very fine and has very good flow properties, which is also supported by the values of the angle of internal friction in the range of 26-28°, which represents a low value. Based on the flow function, the source powder can be classified as a free-flowing particulate material and according to the Hausner ratio, it has a "good" flow character.

The raw material under investigation, existing as a powder, presented a significant challenge due to the presence of a very fine dust fraction. This dust fraction posed a substantial risk to worker safety and potential health hazards during

handling and subsequent processing. Although the powder exhibited favorable flow properties, modification into larger units with good flowability and increased particle size was deemed indispensable.

The created granulates have very similar flow properties to the source powder. According to the flow function value, they represent easy-flowing particulate materials and the Hausner ratio value is the same as for the powder "good". There is a slight difference in the value of the angle of internal friction, which is in the range of 33°–39°, which is caused by the angular shape of the created granulates.

The dustiness of the powder material and the formed granules is completely different after the granulation process. The source material was extremely dusty; the dustiness of the formed granules was minimal.

From a material point of view, the starting powder and the resulting granules have the same chemical composition without additives. This internal consistency of the material enables the direct use of these granules in subsequent additive manufacturing processes

6 CONCLUSION

Based on the experimental findings, the generated granulate exhibits flow properties that are demonstrably as satisfactory as the original powder material. This characteristic translates to its suitability for further material recovery processes. For example, the granulate can be readily utilized for filament creation through extrusion techniques. A critical aspect addressed during the granulation process was the particle size distribution. The initial powder's excessive dustiness and associated dosing difficulties posed a significant challenge for further processing and worker safety. The achieved results demonstrate a successful transformation: the newly formed granules possess both good flow properties and minimal dustiness. This translates to their potential application in a diverse range of additive manufacturing technologies beyond filament extrusion (FFF). Selective Laser Sintering (SLS) represents another avenue for their utilization. Notably, the filament generated from the granulate through FFF extrusion would possess the material characteristics of PA2200. This specific material, PA2200, is lauded for its suitability in producing functional prototypes, tooling, and even final products across various industrial sectors.

7 ACKNOWLEDGMENTS

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8 REFERENCES

[Acevedo 2012] Acevedo, D., Muliadi, A., Giridhar, A., Litster, J.D., Romanach, R.J.: Evaluation of three approaches for real-time monitoring of roller compaction with near-infrared spec-troscopy, *AAPS PharmSciTech* 13 (2012) 1005–1012, DOI:10.1208/s12249-012-9825-0.

[Arndt 2018] Arndt, O.R., Baggio, R., Adam, A.K., Harting, J., Franceschinis, E., Kleinebudde, P.: Impact of different dry and wet granulation techniques on granule and tablet properties: a comparative study, *J. Pharmaceut. Sci.* 107 (2018) 3143–3152, DOI:10.1016/j.xphs.2018.09.006.

[Cohn 1966] Cohn, R., Heilig, H., Delorimier, A.: Critical evaluation of the compactor, *J. Pharmaceut. Sci.* 55 (1966) 328–331, DOI:10.1002/jps.2600550312

[Eckert 2022] Eckert, M.: *Aditivne technologie a reverzne inzinierstvo*. TNUNI (2022), ISBN: 978-80-8075-983-4.

[EPFL 2024] EPFLdatasheet, https://www.epfl.ch/schools/sti/ateliers/wp-content/uploads/2018/05/sls_PA2200_EOS.pdf, last accessed 2024.

[FREEMAN 2020] FREEMAN TECHNOLOGIES, <https://www.freemantech.co.uk/powder-testing/ft4-powder-rheometer-powder-flow-tester>, last accessed 2020

[Gibson 2021] Gibson, I., Rosen, D., Stucker, B., Khorasani, M.: *Additive Manufacturing Technologies*, Springer (2021), ISBN: 978-3030561260.

[Gupta 2005] Gupta, A., Peck, G.E., Miller, R.W., Morris, K.R.: Real-time near-infrared monitoring of content uniformity, moisture content, compact density, tensile strength, and young's modulus of roller compacted powder blends, *J. Pharmaceut. Sci.* 94 (2005) 1589–1597, DOI:10.1002/jps.20375.

[Kozior 2020] Kozior, T.: Rheological Properties of Polyamide PA 2200 in SLS Technology. *Tehnicky vjesnik - Technical Gazette* 27, (2020), DOI:10.17559/TV-20190225122204.

[Krupova 2024] Krupova, H., Sternadelova, K., Mesicek, J., Ma, Q.-P., Hajnys, J.: Experimental evaluation of selectively laser sintered polyamide 12 surface treatment for direct electrodeposition, *Progress in Organic Coatings* 186, (2024), 107968, ISSN 0300-9440, DOI:10.1016/j.porgcoat.2023.107968.

[Malashin 2024] Malashin, I., Martysyuk, D., Tynchenko, V., Nelyub, V., Borodulin, A., Galinovsky, A.: Mechanical Testing of Selective-Laser-Sintered Polyamide PA2200 Details: Analysis of Tensile Properties via Finite Element Method and Machine Learning Approaches. *Polymers* 16, 737 (2024), DOI: 10.3390/polym16060737.

[McAuliffe 2015] McAuliffe, M.A.P.P., O'Mahony, G.E., Blackshields, C.A., Collins, J.A., Egan, D.P., Kiernan, L., O'Neill, E., Lenihan, S., Walker, G.M., Crean, A.M.: The use of PAT and off-line methods for monitoring of roller compacted ribbon and granule properties with a view to continuous processing, *Org. Process Res. Dev.* 19 (2015) 158–166, DOI:10.1021/ops5000013.

[MICROTRAC 2015] MICROTRAC, PartAn3D Dry Particle Image Analyzer Operation and Maintenance Manual, Microtrac, <https://microtrac.com/products/particle-size-shape-analysis/dynamic-image-analysis/partan-3d>, last accessed 2015.

[Palugan 2022] Palugan, L., Moutaharrik, S., Cirilli, M., Gelain, A., Maroni, A., Melocchi, A., Zema, L., Foppoli, A., Cerea, M.: Evaluation of different types of mannitol for dry granulation by roller compaction, *Journal of Drug Delivery Science and Technology*, Volume 75, Sep-tember 2022, 103619.

[Peciar 2017] Peciar, P., Macho, O., Eckert, M., Fekete, R., Katora, P., Juriga, M., Kabát, J., Gabrisová, L., Peciar, M.: Design of particulate material compactor rolls diameter. *Acta Polytechnica* 57 (4), 263-271 (2017), DOI: 10.14311/AP.2017.57.0263.

[Peciar 2018] Peciar, P., Fekete, R., Peciar, M., Macho, O.: The method of granulation of the particulate material, the granulator of the particulate material with the flat matrix. Patent WO 2019/111236 A1, PCT/IB20 18/059800. (2018).

[Salman 2007] Salman, A.D., Houdslow, M.J., Seville, J.P.K.: Granulation, in: S.J.P.K. Salman A.D, M.J. Hounslow (Eds.), Handbook of Powder Technology, Elsevier, 2007, p. 1375.

[Stucker 2016] Stucker, B.: Additive Manufacturing Technologies, Springer (2016), ISBN: 149394455X0

[Sun 2016] Sun, C., Kleinebudde, P.: Mini review: mechanisms to the loss of tabletability by dry granulation, Eur. J. Pharm. Biopharm. 106 (2016) 9–14, DOI:10.1016/j.ejpb.2016.04.003.

[VEXMA 2024] VEXMA, <https://vexmatech.com/pa-2200.html>, last accessed 2024.

[Wagner 2013] Wagner, C. M., Pein, M., Breitzkreutz, J.: Roll compaction of mannitol: compactability study of crystalline and spray-dried grades, Int. J. Pharm. 453 (2013) 416–422, DOI:10.1016/j.ijpharm.2013.05.024.

[Zarybnicka 2022] Zarybnicka, L., Petrů, J., Krpec, P., Pagac, M.: Effect of Additives and Print Orientation on the Properties of Laser Sintering-Printed Polyamide 12 Components. Polymers (Basel) 14, (2022), DOI:10.3390/polym14061172.

Tab. 1: Material Properties of PA2200 Used in SLS 3D Printing

Material Properties	Description
mechanical strength	PA2200 offers high mechanical strength, making it suitable for functional prototypes and also final parts that require significant durability.
flexibility	PA2200 exhibits a certain degree of flexibility, allowing for the production of parts that can be bent without breaking.
heat resistance	PA2200 has good thermal resistance, making it suitable for applications that may be exposed to moderate temperatures up to 150°C.
chemical resistance	PA2200 is chemically resistant to many solvents and chemicals.
hygroscopicity, dimensional stability	PA 2200 has low moisture absorption, which minimizes material expansion and deformation due to changes in humidity. This ensures high dimensional stability, which is important for the production of precise parts and components that must maintain their dimensions in various environments.

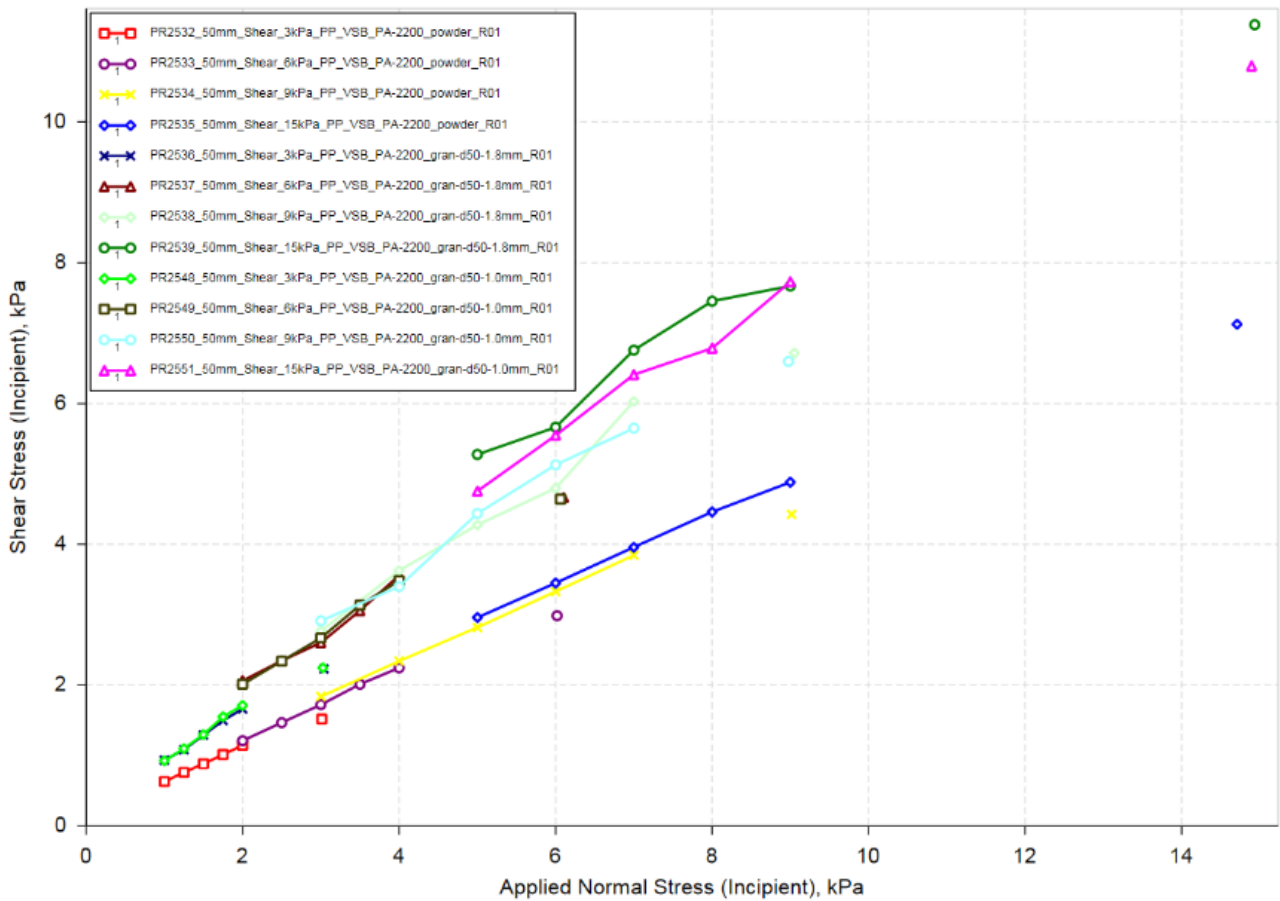


Fig. 4: Measured results of shear tests on the Freeman FT4 device

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