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Modelling of Powder Filling of HIP Canisters

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Abstract. Hot-isostatic pressing (HIPing) of powders is achieved by placing them inside a canister, which is then evacuated and sealed. Canister filling is a critical step. Consistency in powder packing in the canister and increases in packing density will improve process efficiency and the predictability of HIP canister collapse, leading to less wastage. Understanding the effect of powder morphology, properties and characteristics on the can filling process and subsequent compaction is vital to optimizing canister design and the filling system. Conventionally, this has involved conducting numerous costly and time-consuming trial-and-error experiments. Computational modelling offers an alternative optimization path. Discrete element method (DEM) simulation of a powder filling process has been developed by GRI Inc. and its application to the US Department of Energy's radioactive Idaho calcine powders has been investigated. A comprehensive analysis of a non-radioactive simulant powder has been conducted. A DEM model was developed with validation of the model using experimental data obtained from the filling system development program.

Introduction

The HIP technology offers benefits for treating nuclear wastes and is the current baseline option for treating the Idaho calcines [1]. Approximately 4,400 m^3 of highly radioactive Idaho calcine powders are stored in underground silos at the Idaho National Laboratory site [2]. The current plan is to extract the powders, load them into stainless steel canisters and consolidate them via HIPing to reduce the waste volume and form a more stable, dense wasteform suitable for transport and disposal.

Packing radioactive powder into HIP containers offers several challenges. Firstly, maximizing packing density is required to maximize throughput. The canister must be evenly packed so that shrinkage is predictable and controllable, especially radial shrinkage, as the HIPed canister will be placed into cylindrical metal overpack containers. Secondly, the filling system is a connect-disconnect process. It must be designed to control and minimize contamination from radioactive dust while operating remotely to minimize radiation exposure to the technical operators.

Conventionally, the development of bulk materials handling systems involves the construction of prototypes, followed by time-consuming trial-and-error experiments. Non-radioactive simulants of the Idaho calcine have been produced by the US Department of Energy (DOE) for process development trials. The simulant used in this research was created by a fluidized bed calcination process, similar to the Idaho radioactive waste calciner, to produce a powder with the same bulk material properties as the actual calcines. Hence, experimental data can be representative, and the test and design outcomes can be applied to an actual nuclear waste system.

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However, the analysis of nuclear waste and the production of nuclear waste simulants is often expensive and made by specialized, small-scale production facilities. This makes it a scarce material for process validation and technology demonstrations. When a different stream of nuclear waste needs to be assessed, a slight change in the formulation may lead to completely different bulk material properties and behavior. Therefore, preliminary process developments are usually based on limited powder property data, such as particle size distribution and bulk density. Due to its scarcity, the waste simulant is often recycled numerous times to complete a testing program. Repetitive attrition can alter the flow properties of the recycled material, which may lead to results that are no longer representative of the actual nuclear waste. The trial-and-error approach has made process development for nuclear waste treatments challenging and sometimes prevented the development of new technologies.

Computational simulation based on the Discrete Element Method (DEM) offers an alternative approach. DEM is computationally demanding, which limited its industrial application in the past. However, with the advancement in computing hardware and software, DEM is increasingly being used as a tool for industrial process development. Previous studies have shown that a calibrated DEM model can accurately simulate industrial-scale applications [3-5]. This method has also been applied to scarce materials for research and development [6].

This study aims to investigate the potential of DEM for developing process systems that involve handling scarce and hazardous materials, a challenge commonly faced in the HIP industry. This paper presents a validated DEM model, which is employed to simulate an automated HIP can filling process.

Idaho Calcine Waste Simulant

Idaho calcine waste was converted from high-level aqueous radioactive wastes between December 1963 and May 2000 for volume reduction and safer temporary storage at the Idaho Nuclear Technology and Engineering Center (INTEC) [2]. A total of 4,400 m^3 of calcined solids were produced, retrieved and stored in Calcined Solids Storage Facilities (CSSFs). CSSFs are storage vaults consisting of stainless-steel storage bins within a concrete vault. There are seven CSSFs, with five reaching their total capacity. Although the calcined waste management strategy permits the interim waste storage of the Idaho calcine, the long-term plan is to retrieve the calcine, treat the calcine and ship the treated calcine to a long-term storage or disposal facility. Different calcine compositions across the bins reflect the composition of the fuel that was processed. In this study, alumina calcine (the predominant type and likely first to be extracted from the bins) waste simulant was selected as the sample material.

The extraction of the calcines from the CSSFs, their transfer to a treatment facility and loading into the HIP powder handling present challenges. Designing and implementing processes for these activities requires an understanding of the powder's bulk material behavior and chemical, physical and radiological characteristics. Properties of the radioactive calcine retrieved from the storage facility were published in a previous study [7]. Experiments were carried out in hot cells to determine the physical properties of the radioactive calcines, and the results were compared with non-radioactive calcine simulants. The published data includes bulk density, particle size distribution, instantaneous flow function, instantaneous wall friction angle and compressibility. This technical report concluded that there was little significant difference in properties between the simulated and radioactive materials. A more recent alumina calcine simulant has been acquired and characterized to provide certainty of data input to the DEM model.

Flow Property Testing and Characterization of Calcine Simulant

A simulant sample was tested and characterized using a Freeman FT4 powder rheometer (FT4) according to ASTM standards [8]. The waste simulant is an alumina-based material produced by a DOE contractor using a fluidized-bed calcination process similar to that used at INL. The

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composition is based on alumina calcine, minus the toxic elements, as given in Staiger and Swenson [2]. The simulant consists of free-flowing rounded, white, cream and orange-brown colored granules (Fig. 1) with low cohesiveness and excellent aerability. Particle size distribution determined by a Malvern Mastersizer 3000 revealed a bimodal distribution, with D10 = 20.0 μm, D50 = 101 μm, and D90 = 363 μm. Bulk density is found to be 1.21 g/cc. Compressibility and wall friction are shown in Fig. 5 and Fig. 6, respectively.

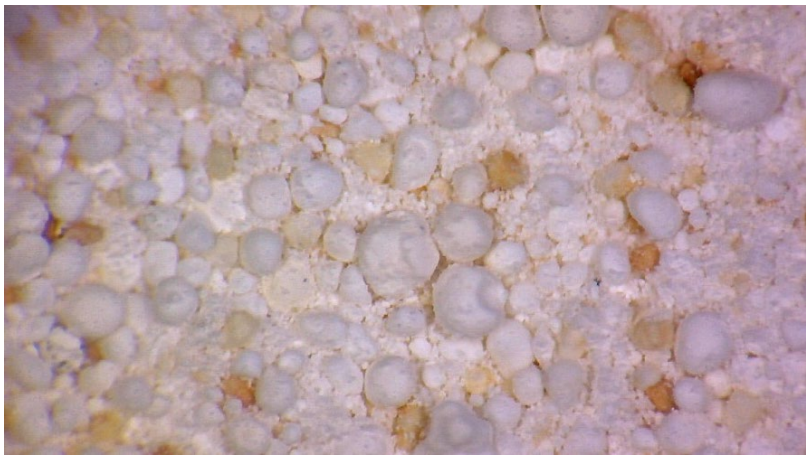

Figure 1: Microscopic image (×10 magnification) of Idaho calcine simulant

Additional experiments were conducted using a Flat Bottom Hopper Discharge Calibration Test Rig (FBH), previously validated to calibrate the DEM model for industrial applications [9]. The FBH was partitioned to have a footprint of 300 × 50 mm before 3.6 kg of simulant was loosely poured into the hopper. After the hopper was filled, the swing gate was opened to discharge the material, via a 15 mm width slot, into a base ring with an outer diameter of 160 mm × inner diameter of 153.5 mm × 50 mm tall, positioned at 250 mm below the hopper. A high-speed camera was used to record the flow of the material. The drained angle of repose was also measured after the discharge was completed.

Despite the capability of FBH to make an angle of repose Θ_R measurement, the pile formation was disrupted by the rapid and unsteady flow towards the end of the hopper discharge test. For better measurement consistency, the angle of repose was determined by the hollow cylinder method [10].

Development of a DEM Material Calibration Model

The interactions of "real-life" particles are complicated with large particle sizes, shapes, surface roughness and charge variances. Agglomeration of granular solids brings further complexities with particle arrangement in the granule, producing surface asperities, intragranular porosity, as well as different adhesion sources and other variables, such as uneven moisture distributions in materials [11]. If the contact model is to be applied at the single particle level, it is necessary to establish the model parameters at the actual particle-particle interaction level. This microstructure method would require considering a highly complex interface interaction situation, including surface topology, chemistry and interstitial media properties.

It is challenging to capture the complex microscale particle-particle contact. Therefore, bulk solid behavior is typically modelled on a mesoscale. With this methodology, each DEM particle represents the mesostructure of the bulk material. The particle-particle and particle-boundary interaction properties can be determined from bulk scale experiments, such as bulk density, compressibility and angle of repose, rather than measuring the microscopic interaction properties, like friction or adhesion surface energy for each particle.

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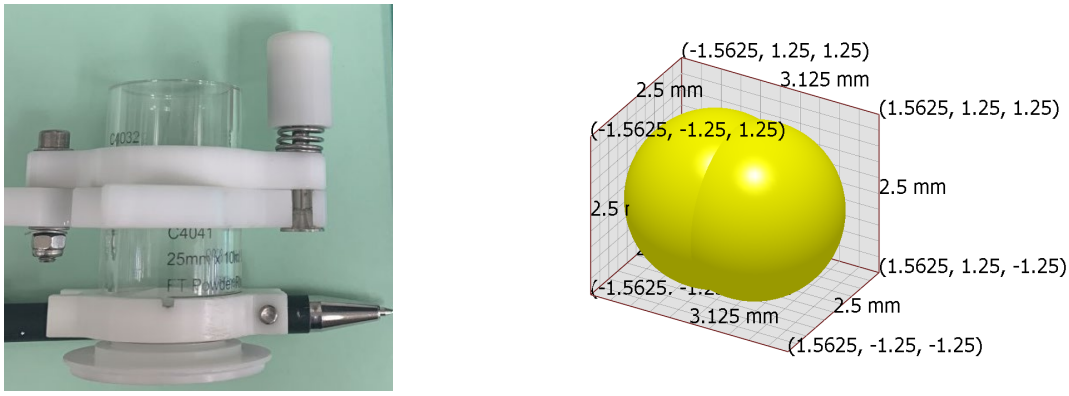
The rationale for this is that DEM simulations are computationally demanding. To accurately model an existing system, it would be excessive to analyze every single particle at life-size, which makes this method prohibitive for any industrial-scale application. Previous studies have shown that even if the actual particle-particle interaction is measured accurately and applied to the contact model, it is still not adequate to simulate the bulk behavior of the material [12,13]. For the process development of industrial-scale applications, researchers are interested in the bulk material's flow behavior and handling characteristics. The purpose of the contact model is to emulate the interactions between the conglomerates to simulate the flow properties of the bulk material and the handling scenario of industrial applications.

In this paper, the Elasto-plastic Adhesion Model (EpAM) [14–18] was selected as the contact model. This contact model was coded to simulate the compressibility and adhesion of bulk materials. The scaling issue caused by the increase in particle size while keeping the size of the experimental apparatus the same is one of the primary sources of error during calibration. The selected particle size (Fig. 2b) is significant compared to the 10 mL Freeman FT-4 split vessel (Fig. 2a). Therefore, instead of replicating the experimental setup of Freeman FT-4 for calibration, compressibility (Fig. 5) and wall friction (Fig. 6) were determined by a Jenike shear tester [19], with the assumption that the material properties measured by these methods are similar and relevant.



(a) (b)

Figure 2: (a) Freeman FT-4 25mm × 10mL (Part# C4041) Split Vessel; (b) Bulk material is represented by multiple-spherical particles with a diameter of 2.5mm and an aspect ratio of 1.25.

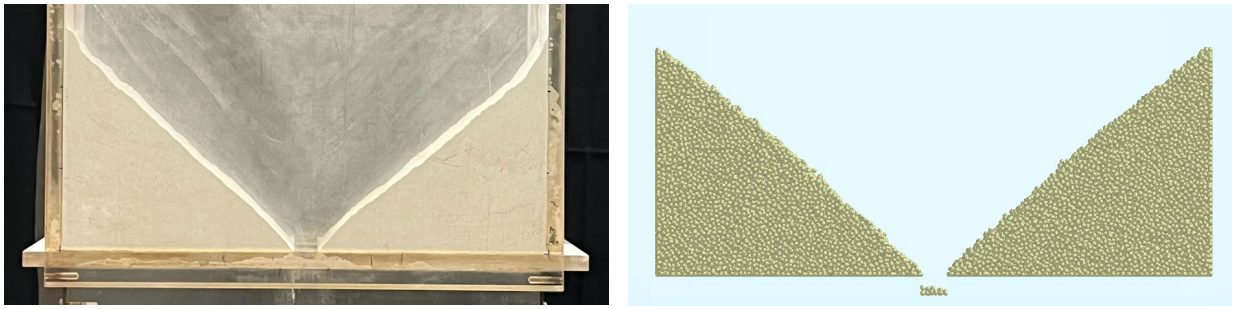
Parameters were selected and adjusted according to a series of virtualized calibration tests processed by Altair EDEM® until realistic and accurate results were obtained. Calibration results are shown in Table 1, Fig. 3, Fig. 4, Fig. 5 and Fig. 6. For general reference, the specification of the custom-built desktop PC is summarized in Table 2. For the capability of running two simulations simultaneously or solving a large model with more than 1 million particles, this PC was configured to have two graphics processors.

Table 1: Calibrated bulk material properties

Parameter	Actual	DEM Simulation
Bulk Density	1.21 [g/cm^3]	1.21g/cm^3]
Drained Angle of Repose (Fig. 3)	38-42°	40-41°
Static Angle of Repose (Fig. 4)	39-42°	40-42°

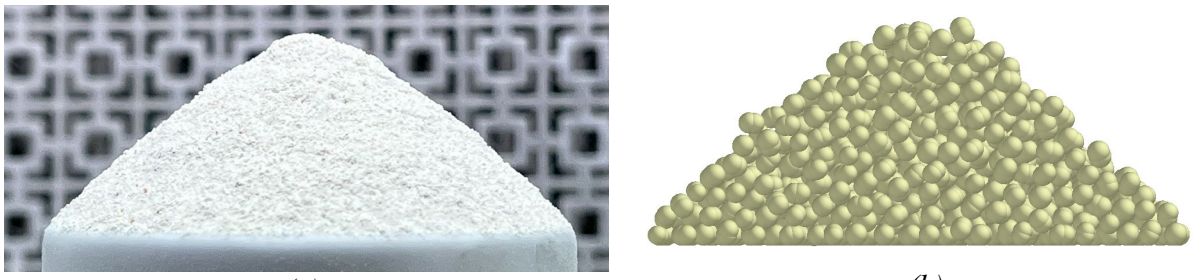
Table 2. Custom-built Desktop PC specification for DEM computational simulations

Operating System	Microsoft Windows 11® Pro 21H2
DEM Software	Altair EDEM v2022.1
Processor	12th Gen Intel® Core™ i7-12700F 2.10 GHz
Graphics Processor	Two × NVIDIA® GeForce® RTX 3080 Ti
Installed RAM	16.0 GB DDR4 3200 MHz
Hard Drive	2TB Crucial® CR2000MX



(a) *(b)*

Figure 3: Flat Bottom Hopper discharge test: (a) actual; (b) DEM simulation



(a) *(b)*

Figure 4: Static angle of repose: (a) actual; (b) DEM simulation

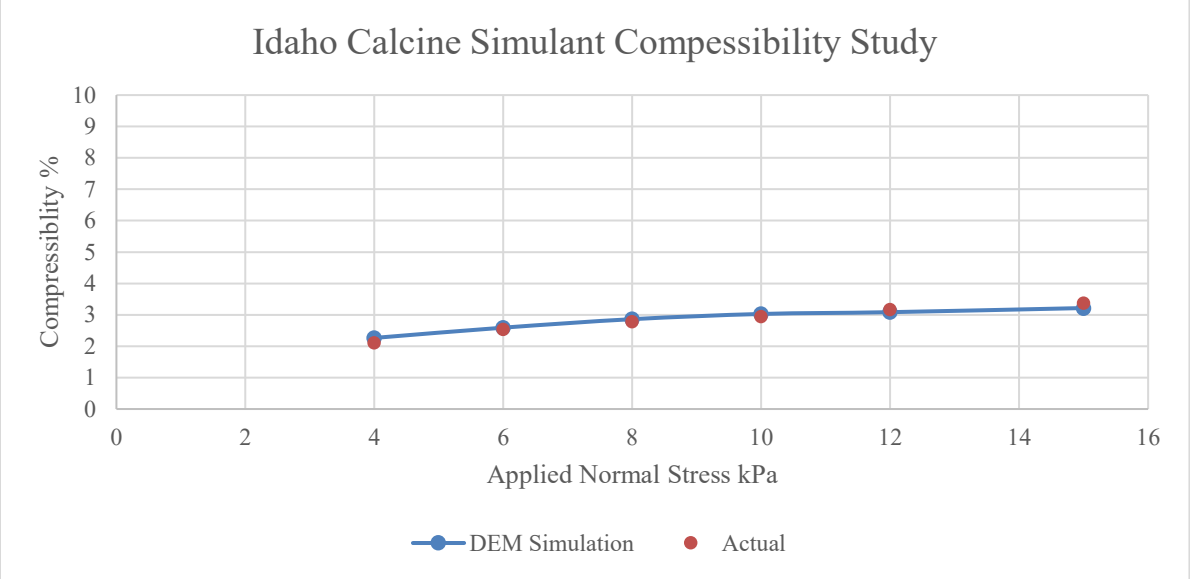


Figure 5: Compressibility of Idaho calcine simulant measured by Freeman FT4 (actual) and Jenike Shear Tester (DEM simulation).

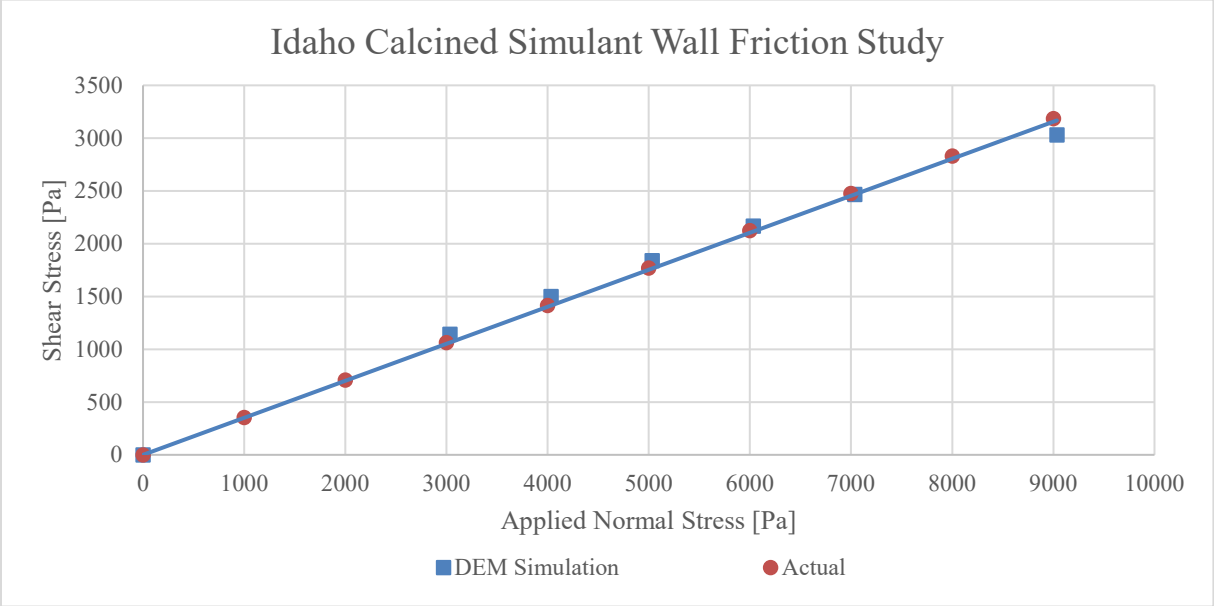


Figure 6: Idaho calcine simulant Wall Friction measured by Freeman FT4 (actual) and Jenike Shear Tester (DEM simulation).

Development of DEM HIP Canister Filling System

After obtaining the calibrated contact model, a three-dimensional DEM model was developed according to the geometry of an integrated HIP canister filling. Two bulk handling scenarios were simulated – standard operation and overfilled scenario.

In the standard operating scenario (Fig. 7), the powder is discharged from the hopper by opening an isolation butterfly valve into the inlet of a rotary valve. The rotary valve rotor (consisting of six pockets) was then rotated +/-180° for 40 cycles to discharge the waste simulant into a container via a unique patented filling nozzle-port coupling system [20]. Material quantity and the pile formation surface profile were examined.

The filling nozzle and process piping were initially filled with materials in the second simulation to simulate an overfill scenario (Fig. 9). After that, the filling nozzle-port coupling system was shut to stop powder flow. The container was then lowered to disconnect. This assessment investigated the amount of residual powder on the top of the filling port. A virtual false floor was placed inside the container, so less particle and simulation time was needed to achieve the overfilled scenario.

Results and Discussions

With the affordable hardware specified in Table 2, simulations of the bulk material handling scenarios were completed in less than five days. The importance of selecting an appropriate time step in DEM simulation was discussed in a previous study [21]. In this research, two identical simulations were set to run at 0.10 and 0.05 Euler time integration to better understand the effect of the time step on the simulated standard operating scenario. The amount of powder received in the container after 40 rotary valve powder dispensing cycles was 2.18276 kg with 0.10-time integration, whereas it was 2.18300 kg with 0.05-time integration. The discrepancy was negligible and within the measurement error of this experiment. As a result, the analysis of the current work continued to use 0.10 as the time step because it was adequate for this application.

In the experiment, 2.197 kg of powder was displaced by 40 rotary valve cycles, which showed that the model could accurately predict the amount of powder dispensed by the rotary valve with an error of 0.65%. Furthermore, the pile formation inside the container closely resembled the experimental observation. The predicted preferential filling towards the left side was verified, but

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with a steeper angle of repose than anticipated. Two additional 40-cycle fills were carried out with vibratory compaction between every 40 cycles to level the surface angle of repose of the pile. Results (Fig. 8) show that the developed DEM model can accurately predict mass flow and hence, cumulative or totalized mass.

(a) (b) (c)

Figure 7: (a) Normal operating scenario; (b) Front view of the container showing the amount of powder received after 40 rotary valve powder dispensing cycles; (c) actual pile formation.

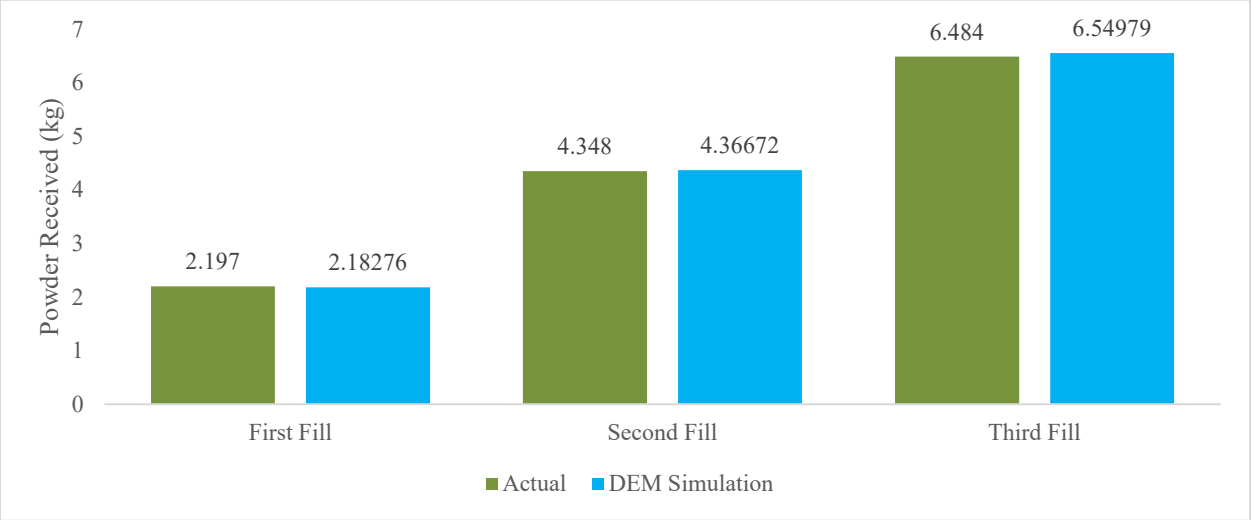

Figure 8: Mass received after 40 cycles (First Fill), 80 cycles (Second Fill) and 120 cycles (Third Fill) of rotary valve discharge cycles.

In the second simulation, the model successfully simulated the fail-safe feature of the filling system. The nozzle-port coupling system could stop the power flow without causing an overspill after removing the container. With a 0.10 time integration, 5.23056g of residual powder was accumulated on the top of the port. Compared to the 6g of residual powder found in the experiment, this is equivalent to an error of 12.8%.

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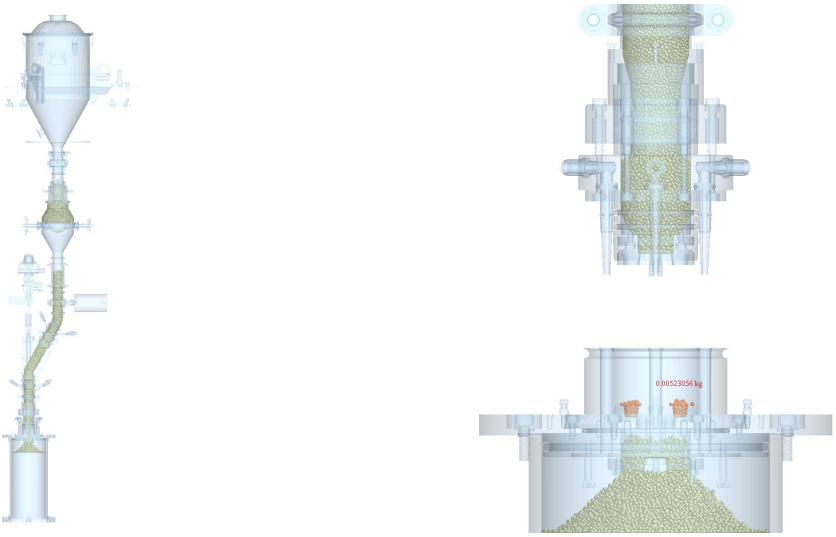

(a) (b)

Figure 9: (a) Overfilling scenario; (b) Fail-safe powder containing disconnection

Conclusion

Predictions made by the calibrated DEM model on the amount of powder dispensed by the rotary valve are quite accurate. Optimization of the model can further reduce simulation time. Therefore, DEM simulation is also a powerful tool for optimizing existing processes. For example, a sensitivity study can be carried out to assess the impact of the pipeline routing on the surface profile of the pile formation. The issue of preferential filling can be resolved by engineering solutions, such as adjusting the filling nozzle's orientation, adding powder flow deflectors, or applying vibratory compaction to level the powder bed.

Powder hold-up above the HIP canister port after an overfilling scenario was predicted by the DEM model to be higher than expected. The overestimation is believed to be caused by the mechanical interlocking issue between the scaled particle and the nozzle outlet. Future research should repeat the experiment with finer particles to investigate if the discrepancy was an issue related to the scaled particle size. Despite the overestimation of powder hold-up, it is insignificant compared to the overall inventory within the powder filling system.

The simulated surface profile of the filled material in the container closely resembles experimental observation but with a steeper angle of repose than anticipated. Future research should consider an alternative calibration method to determine the angle of repose, such as discharging material from a funnel to form a poured angle of repose.

In-situ measurement is found to be challenging in the current experimental setup. Future development should consider non-disruptive measuring methods, such as LiDAR 3D scanning technology and a new container design, to better quantify the surface profile for model validations. The current research can also be extended to investigate the filling of HIP canisters with different geometries to support the development of near-net-shape manufacturing.

In conclusion, DEM simulation can accurately predict a HIP canister filling process when the contact model is adequately calibrated against powder characterization data. The predicted powder discharge quantity and surface profile of the pile formation are valuable design inputs for the preliminary engineering of HIP canister filling systems for producing advanced engineering materials and nuclear wasteforms. These design inputs are particularly beneficial for selecting process equipment and capacity assessment.

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