Critical Review on Engineering Mechanical Quality of Green Compacts using Powder Properties†

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Abstract

For processes involving particulate materials, mechanical properties of green compacts are of great interest when they are final or intermediate products. Optimal quality of green compacts is achieved usually with empirical approaches, i.e., unexpected issues in processes or products’ quality are usually mitigated by time and resource consuming trial-and-error methods. Issues of the powder compaction are commonly observed when there are problems in feed materials or operational conditions even without any substantial change in a formula. Such divergent behavior of particulate materials is especially problematic for product developments and reliable operations. It has been widely accepted hypothesis that properties of particles are determinants of mechanical behavior of powder during compaction and the quality of resulting compacts. With recent developments in nanotechnology, characterization and engineering of individual particles at a microscopic or sub-microscopic scale are now feasible. Leveraging recent technological advancements, there has been a good progress in regard to quantitative understanding of mechanical relationships between properties of particles, particle system and final product. This review highlights the recent developments and gaps in engineering mechanical quality of powder compacts in conjunction with the characterization of particle systems and compaction at multiple scales.

Keywords: powder compaction, green compacts, compact quality, powder properties, particle properties

1. Introduction

Compaction is often employed as a terminal process for manufacturing consumer goods and it affects the quality of final products in the most direct way. Mechanical properties of compacts are especially important in post-production processes such as packaging, transportation, and end-use by consumers. For example, medicinal tablets should not only maintain its integrity throughout packaging and subsequent handling but also provide appropriate disintegration characteristics when administered into human body. Likewise, biomass pellets should preserve their shape and size to ensure desired handling characteristics throughout transportation with minimal crumbliness. On the other hand, cosmetic compacts need to have desired friability for consumer use as well as enough strength so that they do not develop aesthetic defects during shipping and handling. Therefore, achieving and maintaining desired quality of compact is of great importance as a final consumer product. Compaction is also utilized as a critical intermediate process, whose product’s properties are crucial in reliable and efficient downstream processes such as sintering of ceramic or compacted metal powders.

Ideally, compaction behavior and quality should be reasonably predicted during product development process, e.g., formulation, and novel particulate materials development. In reality, producing compact with minimal operational anomalies and desirable quality metrics is very much an art. Manufacturers regularly search for an efficient and sustainable window of operational parameters that will reliably produce compacts’ characteristics satisfying often conflicting quality metrics. Balances between operational maneuver, different mechanical quality metrics, and performance characteristics of compacts are usually achieved by tedious trial-and-error approaches due to the lack of adequate scientific understanding of or a systematic approach to the powder compaction. One of major reasons why compaction operation remains empirical, is because the fundamental understanding of powder compaction is still elusive and, as a result, experiential knowledge plays an essential role. However, the extent, to
which such empiricism can be applied, is limited because of particulate materials’ innate heterogeneity and variability. Novel materials or upstream processes require extended time in identifying optimal process parameters from scratch (Bell, 2005; Merrow, 1988). To address this issue and enable a systematic approach in compaction process and quality control, it is essential to expand our knowledge of the fundamental mechanics of powder compaction and to develop quantitative relationships between feed particulate materials’ properties, densification process parameters, and the mechanical quality metrics of final products.

In addition to insufficient fundamental knowledge of powder compaction, subjective quality metrics of compacts impose challenges to the consideration of the end product quality. Many of quality metrics that are employed by industry have been developed and employed reflecting specific usages of particular products. For example, to evaluate adequate strengths of compacts for logistics or end use, one may use a ‘pass or fail’ test, in which a product is dropped from a certain height to determine breakages. Such tests provide only binary information that may provide limited usage as a quantifiable quality metrics. Similar tests can be re-designed to measure mass and height, at which the product begins to break. Obviously such a quality measurement requires more effort and resources, but it will eventually pay-off when one has the means to predict the quality of products based on quantitative relationships taking properties of feed materials and operational parameters into the consideration. Ideally, these types of tests can be replaced with a set of standardized tests that reflect various scenarios of logistical events, e.g., durability, friability, hardness, and solubility that may be relevant to a specific powder compact product depending on the end use of the compacts.

Another challenge to the powder compaction process is the quantitative powder properties characterization. In regard to properties of particulate feed materials, there are two categories of properties that are relevant and meaningful in our context, namely physical and mechanical properties. Physical properties include bulk density, tap density, particle density, particle size distribution, particle shape, and so on. Even though these are fundamental properties of particulate materials, direct relationships between these and the final product’s properties are difficult to establish because of the stochastic properties of particulate materials, insufficient knowledge on the mechanics of the powder compaction process, and limited understanding of how physical and mechanical properties of the powders affect the powder compaction process and properties of compacts. Nonetheless, because of the dominant importance of mechanical properties of powder en masse in the evolution of mechanical properties of compacts, there have been good amount of studies concerning mechanical aspects of powder compaction especially at the microscopic scale. Reflecting these, this review attempts to assess the current knowledge and to identify subjects, studying which will aid the fundamental understanding of the powder compaction process and will enable a systematic engineering approach in efficient and reliable powder compact production. To that end, this review focuses on the quality metrics’ status quo with emphasis on how fundamental approach can help with predicting and controlling the quality of compacts.

2. Compaction as a powder processing operation

Unit operations involving particulate materials as raw materials, have been well known to be prone to issues that are difficult to mitigate through quantitative engineering approaches (Ennis et al., 1994; Merrow, 1984, 1988). Furthermore, powder compaction is one of less addressed powder processes in spite of its critical role in industrial manufacturing. Powder compaction is utilized as a final production process in various industries including pharmaceutical and nutraceutical tablets, cosmetic consumer goods, biomass pellets, as well as an intermediate process in ceramics and powder metallurgy. Compaction of powders is performed by mechanically pressing powder en masse in dies to form a densified powder mass with enough mechanical integrity that can withstand external loads during expected lifecycle. Green compacts are made typically at ambient temperature through following stages (Fig. 1): 1) Rearrangement of the particles by filling large pores accompanied with the increase in number of contacts (coordination number); 2) packing of particles resulting in decrease in porosity with the formation of localized agglomeration of particles, namely secondary and higher order particle structures; 3) increase in the contact area between particles accompanying elastic deformation of particles; and 4) contact enlargement through plastic deformation of particles (Azami and Khoei, 2006; Bortzmeyer, 1992; Castellanos et al., 2005; Cunningham et al., 2004; Fayed and Otten, 1984; German, 2014; Yi et al., 2001).

Even though these stages of powder compaction have been established for decades, quantitative and fundamental understanding of how these stages contribute to the formation of green compacts is yet to be fully elucidated. This is mainly due to the multiscale nature of the compaction process, which evolves towards different scales of scrutiny, i.e., microscale (single particle, particle-particle interactions), mesoscale (secondary and tertiary particle structures), and macroscale (bulk powder system). Furthermore, due to powder system’s intrinsic stochasticity in particle shapes and sizes in addition to the multiscale
and multiphase nature, powder system is considered to be an inherently complex system (de Gennes, 1999). As a result, many investigations of powder compaction are more or less of troubleshooting nature. For example, issues of powder compaction have been identified by Blumenthal et al. (1997) in regards to powder metallurgy, by Alderborn and Nystrom (1996) for pharmaceutical powder, and by Ennis et al. (1994) for general granular materials.

On the other hand, there are established quantitative models describing powder compaction. Some phenomenological models include Heckel (Ilkka and Paronen, 1993), Kawakita (Kawakita and Lüdde, 1971), and Cooper-Eaton model (Cooper and Eaton, 1962). More physically and mathematically rigorous models describe powder compaction as a macroscopic phenomenon. Such a constitutive model includes Cam-clay models (Dimaggio and Sandler, 1971; Schofield and Wroth, 1968). Unfortunately, existing powder compaction models do not provide fundamental understanding on how underlying particle's properties and particle interactions evolves up to macroscopic behavior of powder system during compaction. Therefore, there exists inevitable limitation when one employs existing powder compaction models in predicting and controlling powder compaction process and compact properties.

3. Green compact quality

This review focuses on mechanical aspects of compact properties among many quality metrics of powder compacts. This section briefly summarizes which mechanical properties of powder compacts are reviewed. A quantified and systematic approach in determining the quality of green compacts is an important step in engineering powder compaction. Accordingly, studies on powder compacts quality metrics are reviewed. Quality metrics of powder compacts include fundamental metrics such as density, strength, hardness as well as secondary or phenomenological properties including friability and other defects of powder compacts (Fig. 2).

One of the most important mechanical properties of compact is strength, i.e., the stress level at which a compact loses its mechanical integrity. Compact strength is related to the constitutive structure of a compact originating from the presence of ingredients, how homogeneously different ingredients are mixed, deposited, and compressed. While the measurement of compact strength is usually performed at the macroscopic level, the origin of defects develop and grow when a compact is subjected to a certain level of mechanical loading, which can often be predicted by relative density distributions. The observation that the corresponding mechanical behavior is closely

![Figure 1](image_url)

Fig. 1 Four stages of powder compaction. In the initial stage (Stage 1) of compaction, rearrangement of the particles by filling large pores accompanied with the increase in number of contacts (coordination number). When the rearrangement of particles completes, volume of powder mass further decreases due to the packing of particles with the formation of localized agglomeration of particles (Stage 2). Powder compaction advances with the increase in the contact area between particles accompanying elastic deformation of particles (Stage 3), then particle-particle contacts enlarge through plastic deformations of particles (Stage 4). As pressure increases further, the bulk density of powder mass gets close to the true density of particles, which leads to the relative density of 1.0. Actual mechanisms of compaction overlaps in real-world compaction and the relative density, at which each stage initiates, varies depending on various particle properties.
related to the heterogeneous relative density distribution of a compact, suggests that the strength of compacts develops in relation to loaded powder system’s characteristic stress distribution development pattern inside a powder system. Since it is not practical to measure stress distribution of powder compacts during the compaction process, compaction behavior has long been described by change in relative density of compact to the true density of particles. Relative density of a powder compact has been one of actively investigated and widely used powder compact quality metrics. Therefore, studies on powder compacts’ density are reviewed first.

In following sections, studies involving powder compacts’ mechanical strengths from a more rigorous perspective are reviewed. In regard to quantitative mechanical properties of powder compacts, their integrity presents two distinctive aspects i.e., development of discontinuity of a compact and localized breakage of powders at the surface of a compact. The former relates more to defects of compacts in a somewhat empirical sense. Considering it wide usage, it is important to recognize empirical quality metrics. For example, mechanical quality metrics of compacts are often described or determined by inspecting a presence of surface defects such as crack or capping. In addition, such mechanical defects of powder compacts often arise from irregular or undesirable mechanical properties of compacts meaning that these can be regarded as indicators of mechanical strength of powder compacts. Moreover, mechanical strength of compacts can be determined in a more empirical way. For example, one can simply conduct a fail or pass test as a result of dropping a product from a certain height. This type of test provides a binary result under very specific circumstances. Recognizing broad usage of such approaches, studies involving mechanical defects of powder compacts are reviewed. Subsequently, studies involving mechanical strengths of powder compacts involving breakage or quantitative mechanical properties, which includes elastic moduli, parameters of powder compaction models, are reviewed. In addition, mechanical strength of a compact’s surface has been widely used as an indicator of mechanical properties of the entirety of powder compacts. Compacts surface’s mechanical characteristic itself is of importance in some cases. Therefore, studies involving hardness tests or surface indentation test are also reviewed.

In addition, the properties of end product, i.e., powder compacts, are often determined in the secondary or tertiary ways, most notably Rattler methods such as friability or durability test. In industry, mechanical strengths of products in compressed powder form, are determined in such empirical ways. For pharmaceutical products and densified biomass, friability or durability test are commonly used to determine the level of mechanical integrity of products when they are subjected to a predetermined sustained mechanical impacts. These type of tests generate quantitative quality metrics, but it is hard to correlate them to fundamental properties of compacts, bulk powder, or particles because those tests represent specific mechanical environments. Nonetheless, considering its popularity and frequent usage, studies involving Rattler tests are reviewed.

3.1 Quality; Density and density distribution

Density is the most basic and fundamental property of powder compacts. The term ‘bulk density’ is often used...
It is typically assumed that powder compact that inherently includes voids between particles. Referring to the densities of ingredients, terms like ‘true density’ or ‘solid density’ are used. The relative density, i.e., ratio between bulk density achieved by compression and ‘true density’ of particles, is often used as a parameter indicating the degree of compaction or compaction behavior. Measurement of bulk density of loose and compressed powders are rather straightforward but ASTM D6683-14 (ASTM Standard and D18.24 Committee, 2014) and ASTM D7481-09 (ASTM Standard and D18.24 Committee, 2009) describe standardized measurement procedures.

Considering that the density of bulk solid is determined by its weight and volume whereas volume of bulk solid changes upon compression, density can be an indicator of a degree of compaction. Therefore, macroscopic volume change of bulk solid has long been used to describe powder compaction or as an indicator of a degree of compaction. Some of most widely used descriptors of powder compaction using macroscopic volume change and applied load includes Heckel, Kawakita, and Cooper-Eaton equations (Alderborn and Nystrom, 1996; Fayed and Otten, 1984). Compressibility, which is based on the relative density of powder as described above, is still widely used as a straightforward descriptor of mechanical behavior of powder in industry as well as researches. For example, Bonaccorsi and Proverbio (2006) measured relative density (termed as green density) as an indicator of compaction quality for metallic powder compaction. Perez-Gandarillas et al. (2015)’s study used relative density change to explain tensile strength gain of compacts for different granulated pharmaceutical formula. Due to the straightforward measurement of relative density, which can be determined using Pycnometer and simple weight measurement with known volume, in addition to the volume change during compaction makes this approach attractive especially from the practical point of view. Similarly, Yohannes et al. (2015) used relative density change in investigating compaction behavior of powders with different particle size distributions focusing on the role of fines. They report that fines do not affect the compressibility and strength past initial rearrangement state and therefore fines can be disregarded in computational modeling using discrete particle approach. These recent studies show the utility of bulk density of powder compaction. However, limitations of macroscopic relative density of powder compaction exist as it lacks an ability to predict or indicate local defects such as crack or capping. It is simply because bulk density is a lumped macroscopic measurement and does not explain underlying mechanism of compaction. This can be complemented by measuring density distribution in compacts.

In addition, density or density distribution of compact often used as measurement to validate compaction model.

For example, Huang and Puri (2000) used density in validation of Adachi-Oka model implemented in a finite element approach (Fig. 3), Borowski (2011) Kadiri et al. (2005) Michrafy et al. (2002), and Sinka et al. (2003) used density in validation of Drucker-Prager model solved with finite element models, and Aydin et al. (1994) and Michrafy et al. (2004) used density in validation of non-linear elastic problem with finite element model by successive incremental solution. In addition, Kong and Lannutti (2000) used density to validate discrete element model.

To this end, determination of density distribution of powder compacts is an important characterization that is necessary in fundamental understanding of powder compaction. Density of compacts can be measured with instrumented compaction unit, which can precisely trace the change of height, therefore density can be determined using known weight of charge. This approach is used in very early studies such as Train (1956) and recent studies such as Michrafy et al. (2003). As one of the earliest efforts of using density distribution in powder compaction study, Train (1956) demonstrated that density of compact is not homogeneous and neighbor of a stationary lower punch wall has higher density compared to upper central region. This difference of density is more pronounced with unlubricated die and attributes to defects of compacts such as capping. Therefore, the relationship between density distribution and defects of compact has been viewed as a quantitative predictor of compact quality.

One of major contribution to the developing density gradient during compaction is die-wall friction. This relationship was investigated by Michrafy et al. (2003). This study shows that friction between wall and die decreases...
while compaction progressed for different types of microcrystalline cellulose powders (PH101, PH102, and PH105) by determining the friction coefficient between compact and die wall using the transmission ratio (applied pressure/transmitted pressure), the transfer ratio (radial pressure/axial pressure) and the aspect ratio (height/diameter of tablet). Small caveat of this measurement is the use of published value for the transfer ratio based on the stress measured at the upper punch instead of mean axial stress at a respective depth. In addition, Michrafy et al. (2003)’s study also shows that measured relative densities are lower than both the Heckel equation, which is an exponential relation between applied stress by a punch and relative density, and a predictive model accounting for the exponentially decaying mean axial stress inside a compact away from the surface, on which a stress is applied. The gap between the measured value and predicted values of Heckel equation increases when the depth increases. This observation suggests that stress decreases exponentially inside a powder compact during compaction. However, quantitative contribution of die wall friction to the compaction behavior remains to be investigated since relative densities are experimentally determined only for unlubricated case.

Alternatively, a destructive indentation hardness can be used in determining compact density. However, indentation hardness test is prone to breaking brittle compacts during experiments. In addition, these methods cannot characterize horizontal variability of compact densities, which arises from the interactions with a die wall. Due to such limitations in regard to the accurate characterization of density distributions, research on this particular subject has been limited in spite of the importance of quantitative measurement of powder compacts’ density distribution as fundamental metrics of powder compaction.

Development of non-destructive (or non-invasive) methods has enabled determining the density of compacts without mechanical disruptions of the test subject. X-ray tomography (including computed tomography, namely CT, or computerized axial tomography, namely CAT), Nuclear magnetic resonance (NMR), Magnetic Resonance Imaging (MRI), and Ultrasound are most widely accepted methods in non-destructive testing of materials (Banhart, 2008).

For example, the development of X-ray tomography has been utilized in quantitative density distribution analysis of compacts. Density distribution of alumina compact was determined using X-ray imaging tracing embedded lead balls (Aydin et al., 1994). Finite element model developed using their measurement demonstrated the contribution of the wall friction to the density distribution. However, there were a substantial disagreement between predicted and measured higher versus lower density regions that are attributed to assumed non-homogeneous internal angle of friction throughout the assembly. This heterogeneity of internal angle of friction of powder assembly is yet to be substantiated. Kong and Lannutti, (2000) utilized X-ray Computed Tomography (CT) to determine variances of density in alumina compacts especially for early compaction stages. To trace compaction, they used tungsten marker, which may have interfered during compaction. Richard et al. (2003) utilized X-ray micro-tomography to investigate packing and compaction of glass beads based on void ratio. Although this is not a direct measurement of compact density, it can be useful approach applicable to other types of particulate materials, whose density can be determined with about 10 μm resolution. Sinka et al. (2004) explored the use of X-ray CT scan in measurement of density distribution in microcrystalline cellulose compacts and showed spatial density distribution can be determined quantitatively. Busignies et al. (2006) also measured a density profile in microcrystalline cellulose compacts using X-ray tomography to study the heterogeneous density of compacts and to investigate a correlation between mechanical properties of compacts and compact densities. However, X-ray tomography does not show clear density gradient inside binary mixture compacts. However, this study reports preferential localization of a specific ingredient implying the origin of difference between mechanical properties of whole compact versus mechanical properties of compact surface, such as indentation hardness. It should be noted that the root cause of such preferential localization of an ingredient is not clarified, which can be a result of less than ideal mixing or segregation during deposition of mixture in a die. Miguélez-Morán et al. (2009) used X-ray CT to characterize roller-compacted ribbons of microcrystalline cellulose showing higher compression in the middle than edges. In addition, they developed correlation between the relative density and localized indentation that follows a log-normal relationship.

Nuclear magnetic resonance (NMR) also has been used in studying density distribution of compacts as shown in Djemai and Sinka (2006) but NMR is not as widely used in this field. Caveats of NMR and X-ray based tomography techniques still exists that they are time-consuming and costly. Additional point to X-ray is that the measurement depends on the chemical composition of the test subject and a specific calibration for subject material is required for different formulations, which can be less than straightforward for industries using multi-ingredients. In another effort, Garino et al. (1995) utilized MRI to determine density variations in powder compacts. In addition, Glass and Ewsuk (1995) used ultrasound to determine relative density of alumina compact. Akseli et al. (2011) used both ultrasound and X-ray CT to determine density distribution of ribbon compacted microcrystalline cellulose in investigation of mechanical properties. They showed that...
3.2 Quality: Mechanical defects

Some of major issues of compacts include defects including crack, capping, and uneven surface (Fig. 4). These defects are more qualitative in nature and often examined with a binary designation i.e., pass or fail. In many cases, local irregularity evolves into defective compacts, it is not straightforward to predict or prevent powder compacts’ mechanical defects without fundamental understanding of powder compaction taking the stochasticity of particulate materials into consideration. This is especially problematic during development of products since it is hard to know the risk of defective products a priori and higher defect rate is found after they are fully formulated. Then this situation requires an extensive investigation relying on trial-and-error approach to identify optimal (or working) recipe of process parameters even without finding out actual causes. As an effort to mitigate this issue, Akseli et al. (2014) presents a relationship between capping and process parameters including formulations. In addition, Kuppuswamy et al. (2001)’s study is an attempt to predict the risk of capping by indentation hardness and observe crack development. Kuppuswamy et al. (2001) investigated detection of formula prone to capping using indentation hardness test attributing capping to the residual die-wall pressure introducing microscopic cracks, which is hypothesized to develop into cracks, therefore capping due to insufficient plasticity. This study suggests that the plasticity of particles is related to localized defect development. This idea is in line with that too much elastic rebound after or during unloading may introduce local defects or failure of compacts.

There have been studies to understand where these defects originate. In studying a relationship between smoothness of surface and mechanical characteristics of compacts, Narayan and Hancock (2003) suggests that brittle bulk powder may indicate their propensity to be vulnerable to cracks or surface defects. It should be noted that Narayan and Hancock (2003)’s study seems to refer to bulk powder’s brittleness or ductility to their failure behavior when compressed. In addition, this study did not investigate the underlying mechanism of how brittleness of compact can be related to cracks or surface defects. However, authors suggest roles of particle size distribution, shape, and bonding interactions in initial packing. In addition, this study also implicitly connects brittleness or ductility of compact (or powder mass under compaction) to the same properties of particles, which would be interesting to find out how these are correlated. Recent advancement of experimental methods, which can determine mechanical properties of sub-micrometer sized test specimens, definitely allow quantitative investigation of particles (Govedarica et al., 2012; Karamchandani et al., 2016).

Related to this subject, Ashby and Hallam (1986) and Ashby and Sammis (1990) report theoretical investigation on how microcracks develop and induce failure in solids. Since powder compacts usually bear defects including microcracks, application of such theories to powder compacts may assist quantitative prediction and control of powder compact’s mechanical integrity. More fundamental origin of compact defects, excessive pores, were reported by Shinohara et al. (1999), in which the voids created from the dimples of alumina particles, grain boundaries, prominently larger particles are proposed as major culprits. Correlation between those major causes and defects in the compacts were reported based on observatory investigation and it is expected that modeling approach that can included such information at particle scale and simulate larger scale compaction behavior will further substantiate this hypothesized origin of defects. In addition, considering alumina particle surface being smooth and spherical, the effect of asperity of more irregular particles on defective compact would be interesting.

In a more recent study, Garner et al. (2014) investigated the mechanism of crack development during ejection of microcrystalline compacts using Druker-Prager model implemented with a finite element method. Their study suggests that microcracks develop during unloading as a mechanism of relieving the radial wall stress. Also surface cracks develops when a compact is exiting die due to abrupt decreases of stresses following the elastic expansion whose rate can be controlled by adding taper at the end of die. Recent advancement of nano-technology and computational power is expected to allow linking approaches taken by Garner et al. (2014) with the observa-

![Fig. 4 Typical compacts with no or common mechanical defects including capping, delamination, and crack.](image-url)
tion of Shinohara et al. (1999), which will elucidate the origin and mechanism of defects development during powder compaction process.

### 3.3 Quality: Strength

Mechanical strength of powder compacts is one of most widely used quantitative metrics (Bonaccorsi and Proverbio, 2006; Hayashi et al., 2013; Krycer et al., 1983a, 1983b; Mazel et al., 2014; Perez-Gandarillas et al., 2015; Russell et al., 2015; Yohannes et al., 2015). Compact's mechanical strength is influenced by many parameters including temperature (Rouèche et al., 2006) or properties of particle including particle size (Narayan and Hancock, 2005) in case of tensile strength. Finer particles tend to result in stronger compacts owing to higher surface area (Alderborn and Nystrom, 1996). However, it is notable that the degree of this increase in compact strength varies for different materials.

Compact strength is largely determined by compression pressure (Fig. 5). A positive correlation between these two are empirically known (Sinka et al., 2009), but exact relationship is yet to be elucidated as there are many additional parameters to determine exact compact strength such as loading rate, amount of charge (therefore the dimension of final compact), etc. A basic approach of determining mechanical strength of medicinal tablets can be found in United States Pharmacopeial Convention (2011). Detailed procedure of mechanical tests are reviewed by Amorós et al. (2008) and Podczeck, 2012. Mazel et al. (2014)'s study is notable since they attempted to link practical engineering strength test to more rigorous Drucker-Prager yield criteria.

Sinka et al. (2009)'s study lists processing parameters affecting eventual strength of compact. Notably, they suggested that the compaction behavior and ultimate properties of compacts originates from particle’s properties. However, Sinka et al. (2009)'s have reported such causal relations in a qualitative way. It is simply because of the daunting numbers of intertwined parameters affecting the compaction process. To be quantitative includes identifying dominant parameters and how large their contribution is and finding out how compaction is happening including the die-wall and loading-rate effects, and possibly mechanics at a scale, in which individual particles are scrutinized. Some specific aspect of processing parameter, such as temperature, has been specifically shown to influence mechanical properties of pharmaceutical compacts York and Pilpel (1973). Nonetheless, this generally accepted conjecture on causal relationship between particle’s properties and compacts’ properties have been actively investigated. For example, Zhang et al. (2014)'s study demonstrated an implicit effects of ingredients on a biomass pellets. Similar result is shown for binders commonly used by Zhang et al. (2003) or major ingredients of pharmaceutical compacts (Akseli, 2009; Pandeya and Puri, 2012).

Furthermore, relationships between surface area of particles and medicinal tablet’s tensile strength have been studied extensively (Fell and Newton, 1970; Jetzer, 1986; Leuenberger, 1982; Riepma et al., 1990, 1991, 1992). Particle’s morphology and ductility of compacts has been reported by Galen and Zavaliangos (2005). Galen and Zavaliangos (2005) examined the anisotropic strength of compacts and attributed the anisotropy especially in the ductile powder compacts to the microscopic architectural structure of deformed particles after the compaction. For example, mechanism is different in each case. For ductile powders, compaction involves the flattening of particles which results in greater strength in the transverse direction due to increased crack deflection that results from greater particle overlap. For the brittle powder, it is proposed that particle fragmentation occurring along the compaction direction weakens strength in the transverse direction. Actual quantification of such a claim was not substantiated but it should be possible now due to the development of nano-technology and microscopy. This idea is investigated further by Wang (2007).

In addition, particle size or particle size distribution’s effect on a compaction process have been actively studied (Jiang et al., 2001; Kaerger et al., 2004; Koynov et al., 2013; Morsi et al., 2006). Effect of particle size on compact strength has been well documented (Khan and Pilpel, 1986; McKenna and McCafferty, 1982). In addition, effect of particle shape on a powder system has been investigated in detail, as well (de Bono and McDowell, 2016; Mittal et al., 2001; Yi et al., 2002; Yi et al., 2001). However, study on the relationship between particle shape and
compact’s properties is scarce, which is thought to be because of the difficulty in quantification of particle shape. Studies on discrete element model require a quantitative and direct description of particle morphology and this need has prompted recent studies on quantitative descriptions of particles (Amberger et al., 2012; Coetzee, 2016; Favier et al., 1999; Garcia et al., 2009; Kruggel-Emden et al., 2008; Křupka and Říha, 2015; Li et al., 2015; Rickman et al., 2016; Wu et al., 2016; Zhao and Wang, 2016).

One of notable properties that has not been studied from this perspective include actual mechanical properties of individual particle and evolution of contacts between particles during compaction in relation to bulk responses of powder en masse during compaction or mechanical properties of resulting compacts. Recently, there have been attempts to investigate mechanical properties of particle with powder compaction using nano-indentation as a mean to determine particle’s mechanical property (Cao et al., 2010; Govedarica et al., 2012). Furthermore Portnikov and Kalman (2015) investigated the effect of temperature on elastic properties individual particles and showed that the effective modulus of elasticity decreases while temperature of particles increases. Unfortunately, this approach is not widely performed probably due to the lack of a clear understanding on how one can use such information in predicting or controlling compaction operations.

In addition, one of the challenges of quantitative characterization of mechanical strength of compact is its stochastic nature originating from stochasticity of particle sizes and shape of powder system. Such distributed nature of mechanical strength of compacts can be characterized using Weibull distribution as shown in Phani (1987). Portnikov and Kalman (2014) established a mathematical model describing the distribution of the effective modulus of elasticity of individual particles including salt, potash, granulated gold nano particles (GNP), zirconium spheres, and glass spheres. Russell et al. (2015)’s study demonstrates how stochastic strength of particle system can be quantitatively investigated using synthetic zeolitic granules whose D50 is 1.75 mm.

There also have been studies on the evolution of coordination number during powder compaction (German, 2014). Nonetheless, knowledge on how coordination number and overall interaction between particles contributes powder compacts’ mechanical integrity and responses is generally lacking. It is expected that efforts to elucidate laws governing the evolution of mechanical properties powder compacts from particle scale based on individual particles and interparticle mechanics will expand leveraging those recent developments.

### 3.4 Quality: Hardness

The indentation hardness has been very widely used in industry as a measure of the mechanical property of powder compacts owing to its straightforward and speedy procedure. Accordingly, many studies used the indentation hardness as a characterization method of mechanical properties of compacts (Bonaccorsi and Proverbio, 2006; Chtourou et al., 2002; Kuppuswamy et al., 2001; Tehrani et al., 2010) as well as an evaluation metrics of a compaction process (Chtourou et al., 2002).

Although the indentation hardness is not a fundamental mechanical experiment procedure, relationships between indentation hardness and mechanical properties have been actively developed and widely accepted. Few examples of such studies can be found in Gent (1958), Gubicza et al. (1996), Oliver and Pharr (1992), and Pavlina and Tyne (2008). It should be noted that, these relationships are specific to tip geometry of an indentor (Fig. 6) and subjected compacts especially in relation to the surface roughness (Laitinen et al., 2013). Furthermore, it is more difficult to establish relationships between hardness measurements and fundamental mechanical properties of materials with inelastic or stochastic mechanical responses (Ma et al., 2009) or when the indentation tip material and subject material have comparable mechanical stiffness.

![Commonly used indentation hardness tip shapes shown from the front (top row) and the shape of indentation shown from the top (bottom row). Indentation hardness is measured by the depth of indentation when subject surface is indented by a tip with specific geometry with a pre-set force.](image_url)
nanoindentation tests on micron-sized silica (SiO$_2$) particles to estimate Young's modulus and Poisson's ratio using the indentation test. Gibson et al. (2015) used a finite element model of the indentor geometry. Taylor et al. (2004) used Duncan-hardness test results. For example, Govedarica et al. (2012) used Sneddon (1965)'s equations which assumes semi-infinite half-space of subject material compared to the indentor geometry. Taylor et al. (2004) used Duncan-Hewitt and Weatherly (1989)'s study based on a semi-empirical fracture mechanics to estimate brittleness based on the ratio of the hardness and elastic modulus based on the indentation geometry of a conventional Vickers Hardness test. Gibson et al. (2015) used a finite element modeling to estimate Young’s modulus and Poisson’s ratio using a nanoindentation tests on micron-sized silica (SiO$_2$) particles. It should be noted that Gibson et al. (2015) assumed non-slip contact, which should be further substantiated as assumptions on contact may have a significant effect especially at the microscopic scales (Briscoe and Adams, 1987; Skrinjar et al., 2005; Thornton and Ning, 1998).

Despite above mentioned caveats, there have been efforts focused on the development of relationship between particle’s properties and powder system’s behavior during compaction or properties of compact. Most notably, Cao et al. (2010) performed AFM nanoindentation on individual pharmaceutical particles, e.g., acetalinophen crystallites, ibuprofen crystallites, sodium acetate trihydrate, microcrystalline cellulose, ascorbic acid, tartaric acid and the hydropropyl methylcellulose. They used film sample for hydropropyl methylcellulose whereas other materials were tested as crystalline particles. Elastic modulus was determined based on the measured stiffness and contact area following the Hertzian contact law. Correlating particle hardness and powder compact hardness, which is also determined by AFM nanoindentation, a quantitative relationship between particle hardness with powder compaction performance is obtained. It should be noted that the powder compaction performance has been determined based on quantitative metrics of mechanical properties of compacts. It is clear that there exist apparent cluster of ingredients resulting compacts with poor or good mechanical quality in relation to the hardness of particles. Cao et al. (2010) study is notable as the first attempt to correlate individual particle’s mechanical properties with the macroscopic powder compacts’ properties. There are plentiful opportunities for further investigation such as appropriate contact mechanics theory for AFM nanoindentation on crystalline particles. For example, due to AFM nano-indentor’s small geometry and unknown asperity of contact surfaces of subject materials, Hertzian contact mechanics may not be appropriate to estimate mechanical properties of particles (Carrillo and Dobrynin, 2012; Johnson, 1987; Kendall, 1987). In addition, degree of crystallinity and effects of crystallographic properties, as Cao et al. (2010) and Willems et al. (1993) assumed for the dominant face of the crystal to play a major role on powder compaction, are yet to be fully understood as actively investigated for cellulose (Pinto, 1999; French and Johnson, 2009; Glasser et al., 2012). Further studies will produce quantitative correlation between macroscopic powder compacts mechanical performance and individual particle’s properties.

Development of AFM also opened other in situ characterization of powder compacts as shown in Miguélez-Morán et al. (2009). In addition, it should be noted that particle interactions have been widely studied using AFM (Cleaver and Looi, 2007; Dobrynin et al., 2013; M. Götzinger and Peukert, 2004; Martin Götzinger and Peukert, 2003; Jones et al., 2003; Kani et al., 2007; Tanaka et al., 2008; Tsukada et al., 2004) but mechanical properties of a single particle has not been studied as actively. This is probably due to the mechanism of AFM’s measurement, which relies on the interactive force between AFM cantilever tip and subject surface including mechanical contact force, van der Waals force, chemical bonding, electrostatic force, magnetic forces, and so forth.

3.5 Quality: Rattler test

Rattler test is originally used to test paving bricks. It was adopted to determine strength of metallurgic powder compacts (Blumenthal et al., 1997). Typical rattler test designates number and size of compacts to be tumbled inside a chamber of predetermined dimension. After a specific number of revolutions at a specified speed, the loss of weight including samples, which are broken during the procedure, is recorded and compared to the original weight of intact samples. Similar approach is used to measure of how green compact maintains its mechanical integrity. Such methods include friability test for pharmaceutical tablets, which is described in (USP29, 2016) and durability test for biomass compacts (Fig. 7), which is described in ASABE Standard S269.4 (2002).

Some studies used durability to control or predict the
quality of biomass compacts (Kaliyan and Morey, 2009; Karamchandani, 2013; Wilson, 2010). Similar approaches were used for pharmaceutical compacts (Vinogradov and Komarova, 1962) using the fundamental idea of friability and not the standardized friability test (Krycer et al., 1983a; Sinka et al., 2009).

Since the friability test procedure’s simple concept and its ability to mimic conditions of handling of compacts, durability or friability provide good quantitative measures of mechanical property of compacts. However, the tertiary nature of the test procedure makes this test to be highly dependent on the dimension of the test device, speed of the rotation, and the size of compacts in one test. Furthermore, the accuracy of this test method is not fully examined. The procedure of durability or friability test is well established and the number of samples and repetition is as well defined. Such dependency of test procedures and equipment for biomass compacts are studied by Temmerman et al. (2006). No similar study is reported for friability of smaller and lighter powder compacts. Furthermore, establishing correlative relationship between friability and other mechanical properties may be of interest to overcome the caveat of this type of test.

4. Summary

As reviewed in this article, studies on powder mechanics research have shown the feasibility of developing predictive relationships between mechanical properties and quality metrics of few types of powders. For example, pharmaceutical tablets are formed using powder ingredients such as filler, binder, disintegrant, and active pharmaceutical ingredient, either by dry blending the ingredients or wet granulation of the powder mix followed by compaction. Pandeya and Puri (2012)’s study reports that a set of mechanical properties of powder en masse, such as springback index, compression index, and bulk modulus, were found most suitable for predicting diametral strength, indentation hardness, and friability of compacts. This was found by developing predictive correlations for tablet quality vs. dry and granulated powder’s mechanical properties that were determined using a medium pressure flexible boundary cubical triaxial tester and mechanical quality metrics of compacts.

In another study, similar approach was applied to biomass pelleting: Karamchandani et al. (2015) demonstrated that ground biomass pellet’s quality can be related to mechanical properties of ground biomass. Especially, this study showed that the fundamental mechanical properties at low pressure range of compaction is capable of predicting properties of compacts produced at much higher pressure, which is thought to be due to the importance of early stage of compaction including rearrangement and elastic responses. This informs the significant importance of measuring fundamental mechanical properties at low pressure range as it is much more practical in industrial laboratory than the measurement at higher pressure range, i.e., over 1 MPa.

5. Looking ahead: The role of microscale interactions

The above-mentioned two studies demonstrate that the quality of compacts can be rationally predicted based on the characterization of feed materials’ mechanical properties. The key to these studies is employing quantitative approaches both in the measurements of quality metrics and characterization of feed materials. This quantitative approach is the first step toward systematically establishing optimal compaction processes to produce compacts with optimal quality; avoiding overshooting production operation parameters as can be the case when using trial-and-error. To achieve this ultimate goal, one needs to understand particle properties’ role in and contribution to the mechanical behavior of powder during densification. The ability to predict behavior of powder during compaction based on mechanical characteristics of individual particles and their interactions will provide a means to implement quality control by design that takes the characteristics of the feedstock into account.

An analytical model, which describes how macroscopic compaction mechanics evolves from mechanics of the underlying scales, is absent largely because of the lack of an adequate method to examine mechanics at the scale where individual particles can be scrutinized. The compaction process evolves towards different scales of scrutiny, i.e., microscale (single particle, particle-particle interactions), mesoscale (secondary and tertiary particle structures), and macroscale (bulk powder system). Accordingly, a research question can be posed as how do a single particle’s properties and particle-particle interactions govern...
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