A NEW ENHANCED SORBITOL: CALCIUM DIPHOSPHATE COMPOSITE AS A DIRECT COMPRESSION EXCIPIENT: A COMPARATIVE STUDY

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INTRODUCTION
Currently, the solid dosage forms have the largest acceptance worldwide and have about 80% of market share because of their versatility, safety, easy handling and good stability to heat and moisture compared to liquid and semi-solid formulations [1]. The solid dosage forms may be prepared mainly by using technologies such as wet granulation, dry granulation or direct compression. Direct compression is the simplest and the most inexpensive method in which tablets are produced directly from a mixture of the active ingredient with suitable excipients. In wet granulation, the active ingredient and the excipients are mixed with a wet binder to form an aggregate which is then dried and passed through sieves to form granules of adequate size for the tableting process [2]. In the dry granulation process, the drug and the excipients are mixed and then passed through rollers and subsequently screened to obtain granules of suitable sizes [3].

Among all the above-mentioned processes, direct compression is the most desirable because it requires few unit operations, has less energy consumption and it provides better stability for moisture, oxidation, and temperature sensitive drugs [4]. However, about 80% of the tablet formulations are made by wet granulation. This is explained by the poor mechanical properties exhibited by most drugs [5].

An excipient intended for direct compression applications should have the following characteristics: excellent compressibility, adequate flow, resistance to segregation, rapid disintegration, low sensitivity to lubricants, scaling-up feasibility, and good dilution potential [6]. The fastest and cheapest way to develop a direct compressible excipient is by co-processing.

Co-processing implies a combination of two or more excipients using appropriate technology to obtain a product with improved tableting properties as compared to their physical mixture. Therefore, the original defects are masked, and the beneficial properties are synergized [7]. The most commonly used technologies to obtain co-processed excipients include spray-drying, agglomeration, hot-melt extrusion and co-precipitation [8]. Co-processing minimizes tablet breakdown due to laminating and capping, which are in turn, attributed to energy built up during the tableting process [6].

In a previous study, a novel sorbitol (SOR) and anhydrous calcium diphosphate (ACD) composite with common excipients used for the preparation of tablets by direct compression such as polyvinylpyrrolidone (Ludipress®), lactose (Cellactose 80®) and microcrystalline cellulose (Prosolv SMCC 90®). This new excipient showed potential for use as a direct compression excipient. This new composite material showed potential for use as a direct compression excipient.

RESULTS
This new excipient offered more benefits in terms of functionality than commercial direct compressive co-processed excipients and showed better compressibility than other commercial excipients and its compaction parameter was ranked third after SOR and Prosolv SMCC 90®. However, this composite material was more susceptible to reprocessing than commercial products. Further, it showed a low lubricant sensitivity due to a combination of a plastic and brittle behavior. Moreover, the loading capacity of poorly compressible materials such as gemfibrozil was comparable to that of commercial direct compression excipients. It also showed the fastest in-vitro dissolution of gemfibrozil, whereas commercial products failed to fulfill the US pharmacopeial requirements.

CONCLUSION
This new composite material showed potential for use as a direct compression excipient.

METHODS
This new agglomerated excipient had better flow, compressibility, and compactibility than the physical mixture of SOR and ACD being the 95:5 ratio. The properties of a new agglomerated excipient containing SOR and ACD (95:5; 80:20, 50:50, 20:80 and 6:94 SOR to ACD ratios [9]). The new agglomerated excipient had better flow, compressibility, and compactibility than the physical mixture of SOR and ACD being the 95:5 ratio, which exhibited the best tabletting properties [10].

Cellactose 80® (α-lactose monohydrate: cellulose powder (75:25), Microcell® (α-lactose monohydrate: microcrystalline cellulose (75:25), Prosolv SMCC 90® (microcrystalline cellulose: colloid silica dioxide (98:2), and Ludipress® (α-Lactose monohydrate Polyvinylpyrrolidone: Crospovidone (93:3.5:3.5) are some of the main co-processed excipients currently available on the market. The goal of this study is to evaluate the particle and mechanical properties of a new agglomerated excipient containing SOR and ACD (95:5 ratio), as compared to commercial co-processed materials namely Prosolv SMCC 90®, Cellactose 80® and Ludipress® and the parent SOR and ACD excipients.

MATERIALS AND METHODS

Materials
Prosolv SMCC 90® (lot 690903220) was obtained from JRS Pharma (Rosenberg, Germany). Magnesium stearate (lot 25654) was purchased from Rio Tinto Minerals (Luzenac Val Chisone SA). Sorbitol (lot 20,140,405) and calcium diphosphate (lot BCU250711) were obtained from Shandong Ruiyang Pharmaceutical Technology (Longwood, USA) and Innophos (Cranbury, NJ, USA), respectively. Gemfibrozil (lot 241303947010) was obtained from Chemo Lugano branch (Lugano, Switzerland), Cellactose 80® (lot 1321) was purchased from Meggle (Wasserburg, Germany), Ludipress® (lot 7103644700), Crospovidone (lot 2912588000) and sodium lauryl sulfate (lot 0012730186) were purchased from BASF (Evionnaz, Switzerland).
Preparation of the sorbitol and calcium diphosphate (95:5) composite

Approximately, 100 g of ACD and SOR were mixed at a 5:95 ratio and wetted with a suitable amount of distilled water (5.5 mL) to form a wet mass. This mass was then passed through a # 14 sieve and agglomerated in a spherizer (Model 1LA7 080-6Y60, Medellin, Colombia). The agglomeration process was performed for 5 min with an angle of 30 degrees and speed of ~100 rpm. The material was then dried at 60 °C for 24 h and passed through a 6mesh sieve.

Particle sizes analysis

Excipients were fractionated on a Ro-Tap sieve shaker (Model, RX29, W. S. Tyler Company, Mentor, OH) using stainless steel 180,150, 125, 106, 75, 44 and 38 µm size sieves, stacked together in the order written (Fisher Scientific Co., Pittsburgh, PA). Approximately, 20 g of the sample was shaken for 10 min. The geometric mean diameter, \( d_g \), was determined from the log-normal distribution plot constructed between the sieve mean diameter and cumulative percent frequency using the Mintab software (v.16, Minitab, Inc, State College, PA).

Particle properties

The moisture content was obtained by the gravimetric method heating the sample at 105 °C for 10 min in an infrared moisture balance (GEHAKA IV 3000). True density was determined using a Helium displacement micro pycnometer (Accupyc II 13340, Micromeritics, USA) with ~2 g of a dry sample. Bulk density was determined on 20 g of material and the tap density was measured on a Tap density analyzer (AT2, Quantachrome instruments, USA) for 400 taps. Porosity (\( \varepsilon \)) was calculated as reported previously [11]. Flow rate was obtained from ~20 g of material that passed through a glass funnel having a 13 mm diameter orifice, and its weight was recorded as a function of time.

Tablet porosity

The thickness and diameter of the tablets were measured with an electronic digital caliper (Titer, 0.01 mm sensitivity). The thickness of the tablets was measured at three different points of the tablet, and the average was reported. The compact volume and porosity were calculated according to their geometry as reported previously [12].

Compressibility analysis

Compacts of ~300 mg were made on a single punch tablet machine (060804 Compac, Indemec, Itagi, Colombia) coupled with a load cell (LGD-10K, Omega Engineering, Inc., Stamford, CT) at 1 and 30 s using a flat-faced 6.5 mm punches and die tooling. Pressures ranged from ~10 MPa to 300 MPa and were measured on a strain gauge (LGD-10K, Omega Engineering, Inc., Stamford, CT). Compacts were analyzed immediately after ejected. The natural logarithm of the inverse of compact porosity was plotted against compression pressure to construct the Heckel plots as reported previously [13]:

\[
\ln(1-x) = k\gamma P + A \quad (1)
\]

Where, \( A \) is the intercept obtained by extrapolating the linear region to zero pressure. The slope (\( m \)) is inversely related to the yield material pressure (\( P_y \)), which is a measure of its plasticity [14]. Thus, a low \( P_y \) (usually values<100 MPa) indicates a high ductile deformation mechanism upon compression. Other parameters such as \( D_k \), \( D_n \), and \( D_s \) are related to initial powder packing/densification, total compact densification and particle rearrangement/ fragmentation at the initial compaction stage, respectively [15]. They were calculated as described previously [11].

Compactibility analysis

Compacts were made as described under “compressibility analysis”. The analysis was performed using a tablet hardness tester (UK 200, Vankel, Manasquan, NJ) and the compact tensile strength (\( MPa \)) was then recorded. The crosshead speed of the left moving platen was 3.5 mm/s. The area under the tensile strength curve (AUCTS) obtained from the Leuenberger model was used to determine the compactibility of the materials [16]:

\[
\sigma_t = T_{max} (1 - \exp (-\gamma P \rho)) \quad (2)
\]

Where, TS is the radial tensile strength (MPa), \( T_{max} \) is the theoretical tensile strength at infinite compression pressure, \( \gamma \) is the compression susceptibility parameter (MPa^(-1)), \( \rho \) is the relative density and \( P \) is the compression pressure (MPa).

Dilution potential (DP)

Gemfibrozil was used as a model drug for direct compression due to its poor compaction properties. Tablets of ~700 mg in weight containing different levels of excipient (0, 5, 20, 50, 90, 95 and 100 %) and a poorly compressible drug (gemfibrozil), were prepared, and their tensile strength was determined. Gemfibrozil and the test excipient were mixed in a mortar and pestle for 5 min and then compressed on a single punch tablet press at 30, 90 and 120 MPa at a dwell time of 1 s. The DP was obtained from the area ratio vs composition plots as reported previously [17].

Lubricant sensitivity (LS)

Lubricant sensitivity was assessed by mixing powders previously passed through a 250 µm sieve with magnesium stearate at a 99:1 weight ratio using a V-blender (Rhiddi Pharma, India) for 5 min. Tablets were prepared using a single punch tablet press at a dwell time of 1 s at a pressure, so a compact with ~ 20 % porosity is obtained. The lubricant sensitivity was expressed as the lubricant sensitivity ratio as reported previously [18].

Reprocessing susceptibility

Biconvex compacts of ~30 % porosity, each weighing about 300 mg and measuring 8.7 mm in diameter were made using an eight station tablet machine (Rhiddi Pharma, India). Compact hardness was measured and converted to tensile strength and tablet pieces were milled and passed through a 250 µm sieve, and subsequently compressed. Compacts were analyzed in a hardness tester, and data were transformed to tensile strength.

Elastic recovery (ER)

Compacts of ~300 mg were made on a single punch tablet press equipped with a flat-faced 6.5 mm diameter tooling at 20 % porosity. Tablet thickness was measured immediately after ejected (0.01 mm sensitivity) and after 15 d of storage. The ER was calculated as reported previously [19].

Compact water uptake and compact disintegration tests

Tablets, each weighing ~300 mg, were made on a single punch tablet press using a 6.5 mm round, flat-faced punches and die set at a dwell time of 1 s. Compression forces were controlled, so compacts of 30- 40 % porosity were obtained. Compacts were stored in a chamber containing distilled water at 25 °C for 15 d keeping a relative humidity of ~100 %. The increase of weight was measured with time and expressed as a percentage. On the other hand, compact disintegration was performed in 1000 mL of distilled water at 37 °C employing an Erweka GmbH disintegration apparatus (39-133-115, Hanson Research Corporation, Northridge, CA, USA) at 30 strokes/ min.

Dissolution studies

A formulation mixture containing 600 mg of gemfibrozil, 25 mg of crospovidone, 32 mg of sodium lauryl sulfate, 17 mg of magnesium stearate and 142 mg of testing excipient was blended on a mortar and pestle. The dry mixtures were then compacted using a single station tablet press (Compac 060804, Indemec, Columbia) at ~75MPa to form cylindrical matrices. The release studies were conducted on an Erweka (DT6-K, Erweka GmbH, Milford, CT) Type 2 apparatus operated at 37 °C and 50 rpm for 30 min. A 900 mL of pH 7.5 phosphate buffer was used as a release medium. Aliquots of 5 mL each, were taken, filtered and diluted to 50 mL with 1N NaOH before measurement. The concentration of gemfibrozil was found by UV analysis (HACH DR500, HACHX Company, Loveland, CO) at 276 nm according to the USP 38 NF 38 specifications.
RESULTS AND DISCUSSION

Powder properties

The powder properties of the SOR: ACD composite and commercial excipients are shown in Table 1, and the morphology features are shown in Fig. 1. The agglomerate had 158 µm in size and was comparable to that of Ludipress®, Cellactose 80® and SOR. In contrast, ACD had the smallest particle size followed by Prosolv SMCC 90®. Further, the agglomerate, SOR, and Ludipress® had the lowest true density, whereas ACD and Prosolv SMCC 90® had the highest values. Commercial materials such as Prosolv SMCC 90®, Cellactose 80® and Ludipress® presented the lowest bulk (0.36, 0.40 and 0.48 g/cc) and tap densities (0.5, 0.5 and 0.7 g/cc), whereas ACD and the agglomerate had the largest values (0.69, 0.51 g/cc and 1.03, 0.94 g/cc, respectively).

Moreover, since SOR and the agglomerate presented a large density and regular shape, they also exhibited the smallest porosity (58.9 and 50.6 %, respectively). This is beneficial to dilute small dose drugs. On the other hand, Prosolv SMCC 90® and Cellactose 80® had the largest porosity. Further, Ludipress®, the agglomerate, and SOR had the largest flow rate due to their more regular shape, smooth surface and high bulk density. Conversely, ACD showed the slowest flow rate. This is mainly attributed to its small particle size and irregular particle shape.

![Optical microphotographs of the agglomerate and commercial excipients](image1)

**Fig. 1:** Optical microphotographs of the agglomerate and commercial excipients
Table 1: Powder properties commercial materials were restested for moisture content and results are shown here

<table>
<thead>
<tr>
<th>Property</th>
<th>Agglomerate</th>
<th>Cellactose 80®</th>
<th>Ludipress®</th>
<th>Prosolv SMCC 90®</th>
<th>ACD®</th>
<th>SOR®</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS (µm)</td>
<td>158±2.1</td>
<td>137±2.9</td>
<td>172±2.1</td>
<td>80±1.6</td>
<td>14.3±1.5</td>
<td>17.2±2.2</td>
</tr>
<tr>
<td>Bulk density (g/cm³)</td>
<td>0.51±0.00</td>
<td>0.40±0.01</td>
<td>0.48±0.01</td>
<td>0.36±0.00</td>
<td>0.69±0.00</td>
<td>0.64±0.00</td>
</tr>
<tr>
<td>Tapped density (g/cm³)</td>
<td>0.94±0.10</td>
<td>0.50±0.02</td>
<td>0.70±0.00</td>
<td>0.50±0.00</td>
<td>1.03±0.04</td>
<td>0.70±0.04</td>
</tr>
<tr>
<td>True density (g/cm³)</td>
<td>1.52±0.00</td>
<td>1.57±0.00</td>
<td>1.52±0.00</td>
<td>1.61±0.00</td>
<td>2.99±0.00</td>
<td>1.55±0.00</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>50.6</td>
<td>75.8</td>
<td>65.1</td>
<td>78.9</td>
<td>76.9</td>
<td>58.7</td>
</tr>
<tr>
<td>Moisture content (%)</td>
<td>1.00</td>
<td>1.1</td>
<td>1.3</td>
<td>2.5</td>
<td>0.20</td>
<td>0.80</td>
</tr>
<tr>
<td>Flow rate (g/s)</td>
<td>19.8±1.3</td>
<td>16.5±1.9</td>
<td>22.5±3.8</td>
<td>12.9±5.0</td>
<td>6.9±1.1</td>
<td>23.9±3.2</td>
</tr>
<tr>
<td>Compressibility (MPa²)</td>
<td>13.3</td>
<td>20.0</td>
<td>22.0</td>
<td>25.0</td>
<td>39.0</td>
<td>15.0</td>
</tr>
</tbody>
</table>

Table 2: Tabletting properties

<table>
<thead>
<tr>
<th>Test</th>
<th>Agglomerate</th>
<th>Cellactose 80®</th>
<th>Ludipress®</th>
<th>Prosolv SMCC 90®</th>
<th>ACD®</th>
<th>SOR®</th>
</tr>
</thead>
<tbody>
<tr>
<td>Py (MPa)</td>
<td>57.6</td>
<td>145.6</td>
<td>239.7</td>
<td>102.7</td>
<td>383.0</td>
<td>71.8</td>
</tr>
<tr>
<td>True density (g/cm³)</td>
<td>1.52</td>
<td>1.57</td>
<td>1.53</td>
<td>1.11</td>
<td>2.99</td>
<td>1.55</td>
</tr>
<tr>
<td>Da (g/cm³)</td>
<td>0.67</td>
<td>0.48</td>
<td>0.52</td>
<td>0.36</td>
<td>0.44</td>
<td>0.67</td>
</tr>
<tr>
<td>Do (g/cm³)</td>
<td>0.39</td>
<td>0.26</td>
<td>0.32</td>
<td>0.23</td>
<td>0.17</td>
<td>0.31</td>
</tr>
<tr>
<td>Db (g/cm³)</td>
<td>0.29</td>
<td>0.22</td>
<td>0.20</td>
<td>0.14</td>
<td>0.27</td>
<td>0.35</td>
</tr>
<tr>
<td>All (g/cm³)</td>
<td>862.4</td>
<td>424.8</td>
<td>360</td>
<td>449.6</td>
<td>260.2</td>
<td>76.8</td>
</tr>
<tr>
<td>SRS (%)</td>
<td>30.2</td>
<td>22.4</td>
<td>58.9</td>
<td>1.1</td>
<td>14.3</td>
<td>49.6</td>
</tr>
<tr>
<td>γc (MPa²)</td>
<td>0.016</td>
<td>0.005</td>
<td>0.003</td>
<td>0.014</td>
<td>0.006</td>
<td>0.029</td>
</tr>
<tr>
<td>T (MPa)</td>
<td>4.1</td>
<td>4.6</td>
<td>2.3</td>
<td>5.5</td>
<td>1.7</td>
<td>4.9</td>
</tr>
<tr>
<td>Compactibility (AUCTS) (MPa²)</td>
<td>268.2</td>
<td>141.7</td>
<td>48.1</td>
<td>34.24</td>
<td>48.9</td>
<td>420.5</td>
</tr>
<tr>
<td>Lubricant sensitivity</td>
<td>0.28</td>
<td>0.17</td>
<td>0.82</td>
<td>0.61</td>
<td>0.00</td>
<td>0.13</td>
</tr>
<tr>
<td>Elastic recovery (%)</td>
<td>1.0</td>
<td>0.04</td>
<td>0.02</td>
<td>0.0</td>
<td>0.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Dilution potential (%)</td>
<td>37</td>
<td>36</td>
<td>40</td>
<td>33</td>
<td>82</td>
<td>40</td>
</tr>
<tr>
<td>Compact water uptake (%)</td>
<td>14</td>
<td>1.0</td>
<td>1.1</td>
<td>2.4</td>
<td>1.1</td>
<td>35.7</td>
</tr>
<tr>
<td>Compact disintegration (min)</td>
<td>6.6±0.6</td>
<td>6.3±1.5</td>
<td>1.5±0.6</td>
<td>&gt;30</td>
<td>&gt;30</td>
<td>8</td>
</tr>
<tr>
<td>Geminibrozil release (%)</td>
<td>86±6.6</td>
<td>53±5.7</td>
<td>48±4.8</td>
<td>35±2.4</td>
<td>28±5.4</td>
<td>17±2.9</td>
</tr>
</tbody>
</table>

Anhydrous calcium diphosphate, b. sorbitol, c. powder yield pressure, d. total compact densification, e. initial powder packing/density, f. total compact densification by particle rearrangement/fragmentation, g. the area under the curve from the Heckel model, h. strain rate sensitivity, i. compression susceptibility parameter j. theoretical tensile strength at infinite compression pressure, k. the area under the tensile strength curve obtained from the Leuenerberger model. Experiments were performed in triplicate. Data are given in mean±SD.

Based on the area under the Heckel curve (AUCHC) it is deduced that the agglomerate and ACD had the lowest and highest powder compressibility, respectively. Usually, plastic materials had a high densification, whereas those with a low Py were less compressible. Compressibility is directly obtained from the AUC of the Heckel curve and showed the agglomerate having the highest compressibility followed by SOR. On the contrary, ACD and Ludipress® showed the lowest compressibility. Therefore, the decreasing trend of plastic deformation upon consolidation followed the order: agglomerate>SOR>Prosolv SMCC 90®>Cellactose 80®>Ludipress®=ACD.

The D₀, Dₐ, and Dₚ parameters, calculated from the Heckel plots, represent the initial packing of the material upon die filling, total packing at low pressures, and degree of powder bed arrangement due to fragmentation at low pressures, respectively. The agglomerate presented the largest densification by die filling, and along with SOR had the total largest densification (D₀) and particle rearrangement in the powder bed at low initial compression pressures. On the contrary, Prosolv SMCC 90® and ACD presented the lowest densification driven by gravity forces. This finding is explained by the low particle size and high cohesiveness between particles. Further, the high densification, particle size and regular shape of SOR contributed to its high rearrangement behavior in the powder bed. Conversely, Prosolv SMCC 90® and Ludipress® presented the lowest rearrangement in the powder bed at initial compression pressures (D₀~0.14–0.20). This is attributed to morphological and surface factors.

Fig. 2: Heckel curves of the agglomerate and commercial excipients. Experiments were performed in triplicate. Error bars correspond to standard deviation.
The fitting parameters obtained from the Leuenberger model are shown in table 2 and tensile strength curves are shown in fig. 3 [20]. Prosolv SMCC 90® had the best compactibility, whereas ACD and Ludipress® showed the lowest values. The agglomerate formed compacts that were stronger than those made of Ludipress®, Cellactose 80® and ACD. The magnitude of difference in compact tensile strength increased with increasing pressures. This change was very small for ACD and Ludipress®. The compressibility parameter ($\gamma_c$) is inversely related to the powder yield pressure, and so does the Heckel slope. For instance, plastic deforming materials such as SOR, the agglomerate and Prosolv SMCC 90® presented the highest $\gamma_c$ values of 0.029, 0.016 and 0.014 MPa⁻¹, respectively. The area under the curve of tensile strength (AUCTS) was used to rank materials according to their compactibility. The trend followed the order: SOR>Prosolv SMCC 90®>agglomerate>Cellactose 80®>ACD=Ludipress®.

Reworking susceptibility

Fig. 5 shows the values of the tensile strength of tablets obtained before and after reprocessing. Usually, reprocessing could have a negative impact on the mechanical properties of plastic deforming materials whereas a brittle deformation might improve the tableting performance. In this case, the agglomerate was highly affected, whereas reprocessing improved the mechanical properties of less plastic deforming materials having a high specific surface area such as ACD and Prosolv SMCC 90®. The latter also contains a 2 % of fumed silica, which is known for having the high specific surface area of ~200 m²/g. The loss of compactibility of the agglomerate could be attributed to the way ACD is embedded in the agglomerate after reprocessing. Before reprocessing, the ACD is incorporated into the agglomerated granules, but after milling, the plastic properties of the SOR component prevailed and had a greater negative impact on tableting performance. Some studies suggested that the work of hardening which is generated after pre-compaction might be the cause for the loss of compactibility [21]. The work hardening implies that after recompression a great amount of defects in the particles and entanglement of new dislocations occurs while being deformed plastically. These defects hinder particles and make plastic deformation more difficult for a subsequent compaction process [22].

Lubricant sensitivity

The lubricant sensitivity is a good indicator of the excipient performance since lubricants form a mixed interface between the particles reducing their binding capacity for the compression process [9]. This effect could be related to the amount of lubricant used, mixing time and the presence of excipients with plastic deformation characteristics [23, 24]. The lubricant sensitivity of the agglomerate and commercial excipients is shown in table 2. The lubricant sensitivity was tested with 1.0 % w/w magnesium stearate. The results depicted in table 2 showed that except for ACD most materials were susceptible to magnesium stearate. This means that magnesium stearate was efficient at coating the surface of particles and prevented the formation of hard compacts. This effect was more pronounced in materials that had a smooth surface such as Ludipress® and Prosolv SMCC 90®. Thus, the lubricant coated the surface of the particles and thereby, restricted the contact points between particles rendering compacts of low strength and hence, caused a high lubricant sensitivity. For instance, the negative effect of lubricants on the tensile strength of ACD was negligible since the formation of new surfaces, free of lubricants was not prevented during compaction. Thus, the film lubricant coating on the ACD surface appears as uncompleted, due to its highly irregular particles having a high surface area in which the lubricant was trapped in the pores [18].

Elastic recovery (ER) and dilution potential (DP)

The elastic recovery depends on the elasticity of the compressed material. It occurs due to the reduction in the bonding surface of the
CONFLICT OF INTERESTS

Declared none

REFERENCES
