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Evaluation of Binders and Plasticizers in Kollidon VA 64-PEG Binder Systems

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ABSTRACT

The binders, plasticizers, and dispersants in a polyvinylpyrrolidone / polyethylene glycol / glycerin binder system for PZT were evaluated. Kollidon VA 64 was investigated as a possible alternative binder to Kollidon 25 in a PZT powder system. The target amount of PEG300 in a Kollidon VA 64 system was predicted to be 15 to 30 wt.% PEG300 based on T_g analysis by DSC. The compaction properties (slide coefficient, cohesiveness, green strength, etc.) were analyzed for Kollidon VA 64 – x PEG300 – glycerin systems. The properties in the range of $x = 0$ to 20 for systems without glycerin and $x = 5$ to 20 for systems with glycerin all exceeded the performance of the baseline Kollidon 25 system, of which VA 64 – 10 wt.% PEG300 – 5 wt.% glycerin with adsorbed moisture was the most promising composition due to a compact cohesiveness of 0.84 at 40 kpsi compared to a baseline of 0.44. The effect of dispersants on the compaction properties of a Kollidon 25 – PEG300 binder system was also analyzed, and the compaction properties were also compared to that of a Aquazol 200 – PEG6000 binder system. The powders with dispersant exhibited comparable performance to the baseline, suggesting good compatibility. The compacts produce with the Aquazol 200 – PEG6000 binder exhibited decreased performance when compared to the baseline.

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EXECUTIVE SUMMARY

The use of appropriate organic binders is essential to the manufacture of high-quality ceramic components. Organic binders are used in the manufacture of PZT 95/5 based ceramics for the Ferroelectric Neutron Generator. This SAND Report describes the development of polyvinyl pyrrolidone (trade name Kollidon), polyethylene glycol, glycerin -based binder. Several plasticizers for Kollidon VA 64 were evaluated and the glass transition temperature of 20 wt.% blends is reported. The glass transition temperature, compaction behavior, green strengths, and thermal decomposition behavior of the Kollidon VA 64-PEG300-glycerin system was explored over a wide compositional range. The properties in the range of $x = 0$ to 20 for systems without glycerin and $x = 5$ to 20 for systems with glycerin all exceeded the performance of the baseline Kollidon 25 system, of which VA 64 – 10 wt.% PEG300 – 5 wt.% glycerin with adsorbed moisture was the most promising composition due to a compact cohesiveness of 0.84 at 40 kpsi compared to a baseline of 0.44. The results presented in this report can be used as a basis for further development of improved organic binders to support the fabrication of ceramic components.

This report details the Undergraduate Student Co-op project for Catherine Colletti from her time with the Active Ceramics Value Stream from January to August of 2021.

ACRONYMS AND TERMS

| Acronym/Term | Definition |
|--------------|--|
| α | Compressibility coefficient |
| η | Slide coefficient |
| C | Compact cohesiveness |
| Tg | Glass transition temperature |
| Tm | Melting temperature |
| PVP | Polyvinyl pyrrolidone |
| PEG | Polyethylene glycol |
| PEG300 | PEG average molecular weight (MW) of 300 g/mol |
| PEG6000 | PEG average molecular weight (MW) of 6000 g/mol |
| PZT | Lead zirconate titanate |
| PZT 95/5 | PZT with a composition near the $95 \text{ PbZrO}_3 - 5 \text{ PbTiO}_3$ morphotropic phase boundary |
| IPA | Isopropyl alcohol, 2-propanol |
| TGA | Thermogravimetric analysis |
| DSC | Differential scanning calorimetry |

1. BACKGROUND

Binders are essential in ceramic processing as they hold the powder particles together until the sintering step. Binder systems can contain a complex assortment of organic additives, but the constituents of interest for this powder system are binders, plasticizers, dispersants, and lubricants. The binder adheres the particles, while the plasticizer modifies the binder to make it more plastic and less brittle. Dispersants control particle interactions to reduce agglomeration and reduce the viscosity of ceramic slurries. Lubricants reduce the forces needed to form a ceramic green body. The binder directly affects the workability and formability of ceramic powder, in terms of how the powder flows, compacts, and interacts with both the die and other powder particles.

The glass transition temperature (T_g) is the temperature at which the mechanical behavior of a polymer transitions from brittle to plastic. The glass transition of a binder can be lowered by the addition of a plasticizer. Water in many cases will also act as a plasticizer, so the moisture content of a powder system affects how well plasticized it is. The two main binders used in this study are Kollidon 25 (polyvinylpyrrolidone; PVP) and Kollidon VA 64 (polyvinylpyrrolidone-polyvinyl acetate copolymer; VA64). Kollidon VA 64 is theoretically more plastic, due to a lower glass transition temperature, and less moisture sensitive (Figure 2).

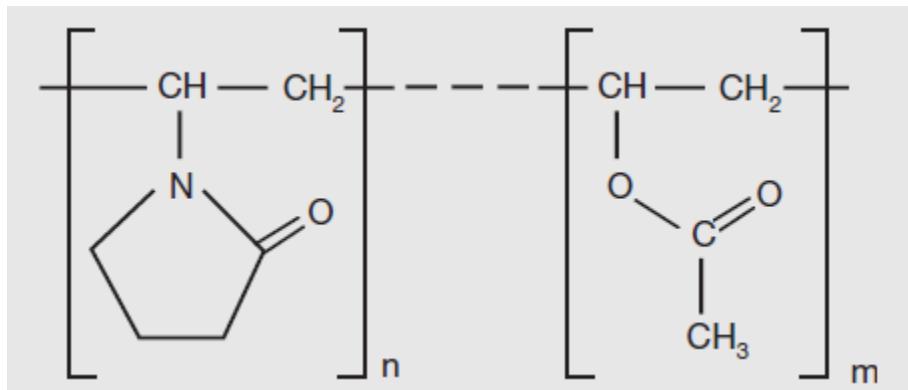


Figure 1. Structural formula of Kollidon VA 64 (copovidone), polyvinyl pyrrolidone – polyvinyl acetate co-polymer.[1]

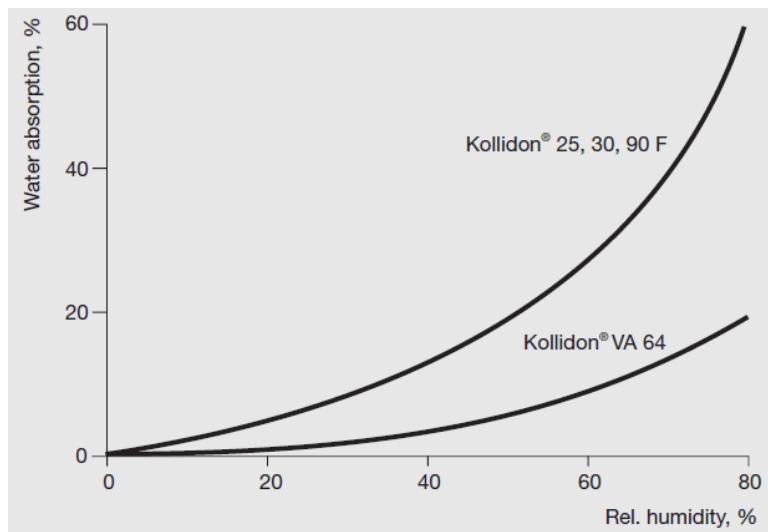


Figure 2. Moisture absorption behavior of Kollidon 25 and Kollidon VA 64.[1]

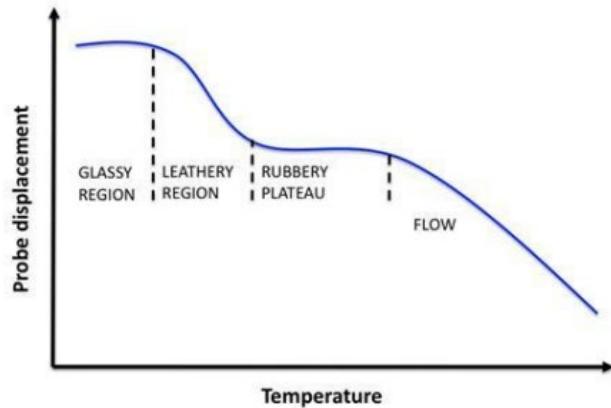


Figure 3. The glass transition temperature occurs between the glassy and leathery region.[2]

The ceramic forming technique used for processing the powder is dry uniaxial pressing to a maximum pressure of 6kpsi, then isostatic pressing to some higher pressure which is determined experimentally. The forming process takes place at room temperature, so the glass transition temperature should be just above room temperature for the optimum behavior of the powder.

2. PROCEDURE

2.1. T_g Determination via DSC

Samples of various polymer blends containing Kollidon VA 64, Kollidon 25, and polyethylene glycol, avg MW 300 (PEG300) were prepared in about 40g of 2-propanol. Polymer blends of Kollidon VA 64 : x PEG300 and Kollidon 25 : x PEG300 were prepared for x = 0, 10, 20, 25, 30, 35, 40, 50. Additional blends were made with an 80:20 blend of Kollidon VA 64 to the various plasticizers shown in Table I. The 10g samples were mixed and heated over a hot plate at 70°C until homogeneous. The samples were dried overnight in a vacuum oven (Model HPP110, Memmert, Schwabach, Germany) at 70°C and 100mbar. The samples were stored in glass vials before Differential Scanning Calorimetry (DSC; Model Q2000 V24.11 Build 124, TA Instruments, New Castle, DE) analysis. The heating program ramped at 10°C/min from -90°C to 250°C. T_g measurements were taken on heating during the second heat; the onset value was used. At least two trials of each sample were taken.

Table I. List of plasticizers evaluated.

| |
|---------------------------|
| Polyethylene glycol 300 |
| Benzyl butyl phthalate |
| Tetronic 1107 Prill |
| Glycerin |
| Propylene glycol |
| Diethyl phthalate |
| Triethyl citrate |
| Triethylene glycol |
| Kolliphor P 188 |
| Kolliphor P 407 |
| Polyethylene glycol 6000 |
| Bis(2-ethylhexyl) adipate |
| Fish oil from menhaden |
| DisperBYK 118 |

2.2. Kollidon VA Binder System

Lead zirconate titanate powder with a Zr/Ti ratio of 95/5 (PZT 95/5; Lockheed Martin Corp, Syracuse, NY) was used as the ceramic powder. The organic additives are summarized in Table II. The binder system in each composition was a mixture of Kollidon VA 64 (VA64), a 6:4 copolymer of polyvinylpyrrolidone : vinyl acetate, and PEG300 (PEG), totaling 4 wt.% addition of the PZT powder. For some slurries, glycerin was also added into the binder as a plasticizer and humectant; 5 wt.% of the total binder was glycerin and the remaining binder was a ratio of Kollidon VA 64 to

PEG300, as depicted in Figure 4. Stearic acid was used as a lubricant in all powders. A baseline powder was prepared with 4 wt.% addition binder in a 70:30(wt.%) ratio of Kollidon 25 to PEG300. A second baseline powder was prepared with the same binder composition and a microcrystalline cellulose pore former, Avicel PH105.

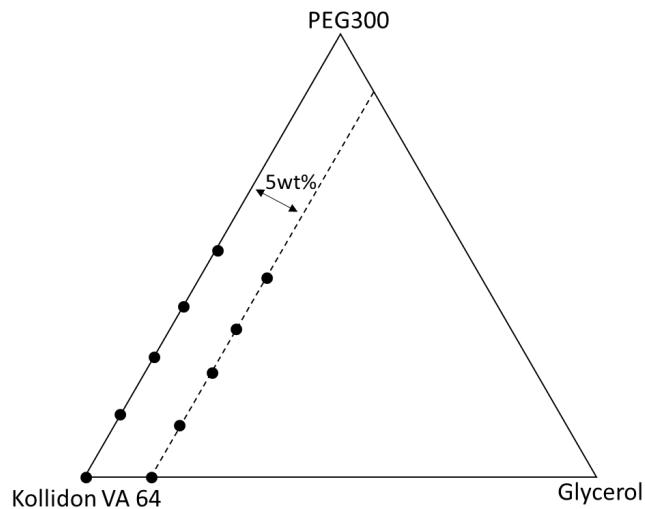


Figure 4. Ternary diagram showing the binder compositions evaluated.

Table II. Summary of Kollidon VA 64 Compositions

| Sample | Kollidon VA 64 (%) | PEG300 (%) | Glycerol (%) | Binder +Plasticizer (wt.%) | Stearic Acid (wt.%) | Pore Former (wt.%) |
|---------------------------|--------------------|------------|--------------|----------------------------|---------------------|--------------------|
| Baseline | 70 | 30 | 0 | 4 | 1 | 0 |
| Baseline + PF | 70 | 30 | 0 | 4 | 1 | 1.25 |
| VA64-0%PEG300 | 100 | 0 | 0 | 4 | 1 | 0 |
| VA64-10%PEG300 | 90 | 10 | 0 | 4 | 1 | 0 |
| VA64-20%PEG300 | 80 | 20 | 0 | 4 | 1 | 0 |
| VA64-30%PEG300 | 70 | 30 | 0 | 4 | 1 | 0 |
| VA64-40%PEG300 | 60 | 40 | 0 | 4 | 1 | 0 |
| VA64-0%PEG300-5%Glycerin | 95 | 0 | 5 | 4 | 1 | 0 |
| VA64-10%PEG300-5%Glycerin | 85.5 | 9.5 | 5 | 4 | 1 | 0 |
| VA64-20%PEG300-5%Glycerin | 76 | 19 | 5 | 4 | 1 | 0 |
| VA64-30%PEG300-5%Glycerin | 66.5 | 28.5 | 5 | 4 | 1 | 0 |
| VA64-40%PEG300-5%Glycerin | 57 | 38 | 5 | 4 | 1 | 0 |

The PZT powder was prepared by burning out organic materials remaining from a previous experiment at 700 °C for one hour (Model 1216 FL, CM Furnaces, Bloomfield, NJ). It was then pre-milled with a disc mill (Model DM400, Retsch, Haan, Germany) with gap spacing of 0.20mm, and ball-milled for twenty-four hours in 2-propanol (20vol.% solids loading) at ~60rpm with 5mm YSZ spherical media. The slurry was pan-dried at 80 °C in an ignition free oven (Model HS-3802-G, TPS, New Columbia, Pennsylvania) until dry, then sieved with a #40-mesh sieve before being suspended in a slurry. Slurries were prepared with a mixer with shear mixing blade (Model Eurostar 40, IKA, Staufen, Germany) by solubilizing all organic materials in 2-propanol (25vol.% solids loading) at 500-600rpm, waiting five minutes to ensure homogeneity, then mixing in 200g PZT powder at 1800rpm until dispersed. Heat was applied via a hot plate to dissolve the stearic acid, then turned off. The slurries were dried overnight in an oven (Model HS-3802-G, TPS, New Columbia, Pennsylvania) at 80 °C, then ground with an automatic mortar and pestle (Model RM200, Retsch, Haan, Germany) until the powder passed through a #40-mesh sieve.

Compaction properties of the powders were measured using a Powder Test Center (PTC; Model PTC-03DT, KZK Powder Tech Corp., Hereford, MD) for both dried powder and humidified powder. The dried powder was left in a vacuum oven (Model V0101, Memmert, Schwabach, Germany) at 60 °C until testing to dry the powders and prevent water adsorption. The humidified powder was conditioned in a climate chamber (Model HPP110, Memmert, Schwabach, Germany) at 25 °C and 40 % relative humidity overnight. Compaction testing took place at room temperature with a 60 s hold time at both 6 kpsi and 4 kpsi maximum pressures. A ½" diameter WC die with hardened steel punches was used to compact the powders. Three samples of each powder at each moisture content and pressure were tested. The moisture content of the powder samples was confirmed using a moisture balance (Model MB120, Ohaus, Parsippany, New Jersey) at a drying temperature of 105 °C and < 1 mg / 60 s end condition.

2.3. Dispersant / Alternate Binder

Four powders with different binder systems, compositions given in Table III, were prepared, and tested for compaction properties following the same procedure as for the Kollidon VA 64 powders. The same Lockheed Martin Corp. surrogate voltage bar material from part one was used; however, the powder samples were ball-milled separately. Each powder sample contained 250g of PZT. Three of the samples had a Kollidon 25 - PEG300 binder with stearic acid lubricant (the baseline composition), and one was composed of Aquazol-PEG6000 with oleic acid lubricant. A hot plate was used to dissociate the PEG6000, then the Aquazol. The PEG6000 did not begin to dissociate until the solvent temperature exceeded 40 °C, as measured by a handheld IR thermometer. The mixing speed was increased from 600 rpm to 1200 rpm during the Aquazol addition to fully dissolve it. Two of the Kollidon 25 - PEG300 powders compositions were also prepared with dispersant, which was added with the other organics when the slurry was prepared. While the Baseline composition for both the Kollidon VA 64 comparison and this part is identical, the PZT powders were ball-milled separately, so an additional Baseline composition was prepared for comparison to the compositions prepared as part of this series.

Table III. Dispersant/Alternate Binder Compositions

| Sample | Binder [†] | | Plasticizer [†] | | Lubricant [†] | | Dispersant [†] | |
|-----------------------------------|---------------------|----------|--------------------------|---------|------------------------|----------|-------------------------|----------|
| Baseline | Kollidon 25 | 2.8 wt.% | PEG300 | 1.2wt.% | Stearic Acid | 1.0 wt.% | - | - |
| Baseline-DisperBYK | Kollidon 25 | 2.8 wt.% | PEG300 | 1.2wt.% | Stearic Acid | 1.0 wt.% | DisperBYK-2155 | 1.0 wt.% |
| Baseline-Carbosperse | Kollidon 25 | 2.8 wt.% | PEG300 | 1.2wt.% | Stearic Acid | 1.0 wt.% | Carbosperse K-XP22B | 1.0 wt.% |
| Aquazol-PEG6000 | Aquazol 200 | 3.0 wt.% | PEG6000 | 0.5wt.% | Oleic Acid | 1.0 wt.% | - | - |
| [†] wt.% addition to PZT | | | | | | | | |

3. RESULTS

3.1. T_g Determination via DSC

The trends for T_g depression of Kollidon VA 64 and Kollidon 25 with increasing plasticizer content can be found in Appendix A. The strength of the various plasticizers evaluated can be seen in Table 4. Several of the plasticizers used formed polymer blends that were milky in color, indicating that they are likely immiscible with the other components; these included Tetronic 1107 Prill, Kolliphor P 188, Kolliphor P 407, and polyethylene glycol 6000.

Table IV. Glass transition and melting temperatures for the Kollidon VA 64 – 20 wt. % Plasticizer mixtures.

| Plasticizer | Lower T _g (°C) | Upper T _g (°C) | T _m (°C) |
|---------------------------|---------------------------|---------------------------|---------------------|
| Benzyl butyl phthalate | 12.22 | - | - |
| Tetronic 1107 Prill | 62.34 | - | 45.69 |
| Glycerin | 14.11 | - | - |
| Propylene glycol | 48.57 | 104.67 | - |
| Dioctyl phthalate | -1.94 | - | - |
| Triethyl citrate | -3.54 | 99.97 | - |
| Triethylene glycol | -12.61 | - | - |
| Kolliphor P 188 | -† | - | 50.37 |
| Kolliphor P 407 | 67.59 | - | 52.48 |
| Polyethylene glycol 6000 | -† | - | 58.835 |
| Bis(2-ethylhexyl) adipate | 56.67 | - | - |
| Fish oil from menhaden | 79.91 | - | -2.095 |
| DisperBYK 118 | 64.68 | - | - |
| PEG300 | 16.29 | - | - |

†No T_g was present, although it may have been obscured by the melting point.

3.2. Kollidon VA Binder System

All the compacts were chalky, with edges prone to small chips. The dried VA64 - 0% PEG300 and VA64 - 10% PEG300 at 40 kpsi were both brittle and broke in half due to handling. Similarly, all dry VA64 - 0% PEG300 - 5% Glycerol and most dry VA64 - 10% PEG300 - 5% Glycerin dry compacts at 40 kpsi contained visible delamination cracks as in Figure 5, so no crush testing for green strength could be performed. No cracking was observed in any of the compacts at 40 kpsi when humidified. The radial and diametral green strength was below the PTC's measurement threshold for several compositions at 6 kpsi.

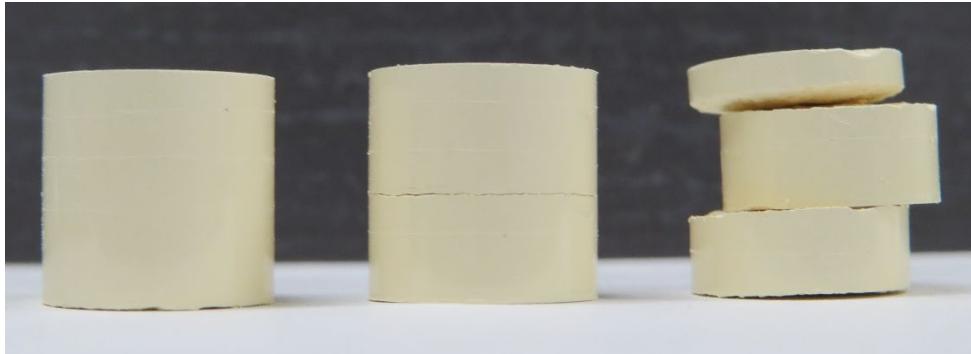


Figure 5. Image showing delamination of the Kollidon VA 64 – 0 wt.%PEG300 compacts.

The compaction properties (compact cohesiveness, compactibility coefficient, slide coefficient, axial and radial expansion, ejection pressure, stripping pressure, green density, and green strength) can be found in Appendix B and Appendix C. The particle size distribution can be found in Figure 6 and Table V.

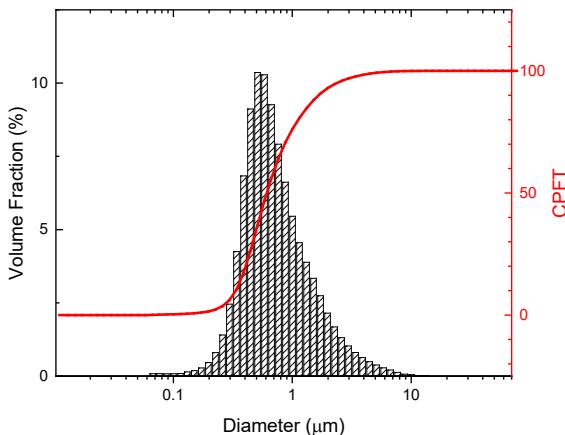


Figure 6. Particle size distribution of the ball milled PZT 95/5 used for the Kollidon VA 64 – PEG – Glycerin study.

Table V. Particle size of the milled PZT 95/5 used for the Kollidon VA 64 – PEG – Glycerin study.

| Parameter | Value |
|-----------------------|-------------|
| D _{v10} (μm) | 0.33 ± 0.01 |
| D _{v50} (μm) | 0.61 ± 0.01 |
| D _{v90} (μm) | 1.67 ± 0.12 |

3.3. Dispersant / Alternate Binder

The viscosity of the slurries was not measured; however, during slurry preparation the Baseline slurry splattered more than the two slurries with dispersant, indicating the dispersants reduced viscosity.

As with the VA64 series, all compacts were chalky with minor edge chipping on handling. The Aquazol-PEG6000 dried compacts at 40kpsi exhibited delamination cracks but did not disintegrate, so crush testing was still performed. Radial and diametral green strength could also not be measured for several compositions at 6 kpsi, as the strength was below the measurement threshold of the equipment (20 N).

The compaction property data is contained in Appendix D and Appendix E. The particle size distribution can be found in Figure 7 and Table VI. Particle of the ball milled PZT 95/5 used for the Alternate Binder / Dispersant study.

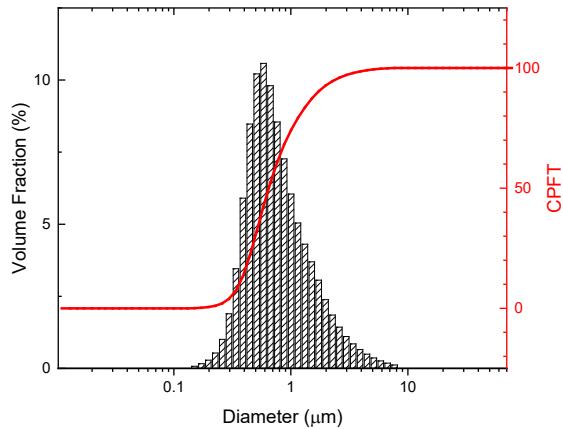


Figure 7. Particle size distribution of the ball milled PZT 95/5 used for the Alternate Binder / Dispersant study.

Table VI. Particle of the ball milled PZT 95/5 used for the Alternate Binder / Dispersant study.

| Parameter | Value |
|-----------------------|-------------|
| D _{v10} (um) | 0.36 ± 0.00 |
| D _{v50} (um) | 0.65 ± 0.00 |
| D _{v90} (um) | 1.70 ± 0.01 |

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4. DISCUSSION

4.1. Glass Transition Temperature

The Fox Equation[3] was used to determine the theoretical T_g of the binders with increasing plasticizer. The glass transition temperatures for Kollidon VA 64, Kollidon 25, and PEG300 were assumed to be 105°C,[1] 155°C,[1] and -77°C,[4] respectively.

The T_g of the Kollidon VA 64 was very similar to the expected values based on the Fox Equation, as seen in Figure 6. The discrepancies from the expected values are likely due to water absorption. The samples were not kept in a dry environment prior to testing and may have picked up varying amounts of moisture which would lead to a lower T_g . As the target T_g is about room temperature, compositions in the VA64-(15-30wt.%)PEG300 range are optimal when there is some moisture content. Based on the plasticizer series, the strongest plasticizers for the Kollidon VA 64 binder are benzyl butyl phthalate, glycerin, dioctyl phthalate, triethyl citrate, and triethylene glycol, while Tetronic 1107 Prill, Kolliphor P 188, Kolliphor P 407, and polyethylene glycol 6000 are incompatible.

All Kollidon 25 T_g values were about 30-50°C lower than they should have been. This is due to an error in the DSC. A sample of Kollidon VA 64 tested in the DSC with those samples was also significantly lower than the same dried Kollidon VA 64 sample tested a month prior. The trend is assumed to be accurate but not the magnitude of the data. At this point, due to the discrepancies, DSC evaluation was halted. For the Kollidon 25 series, an upper and lower T_g was noticed for the polymer mixtures with 30 – 50 wt.% PEG300. While the presence of two T_g 's can indicate immiscibility, the system is fully miscible and a second T_g has been previously reported.[3]

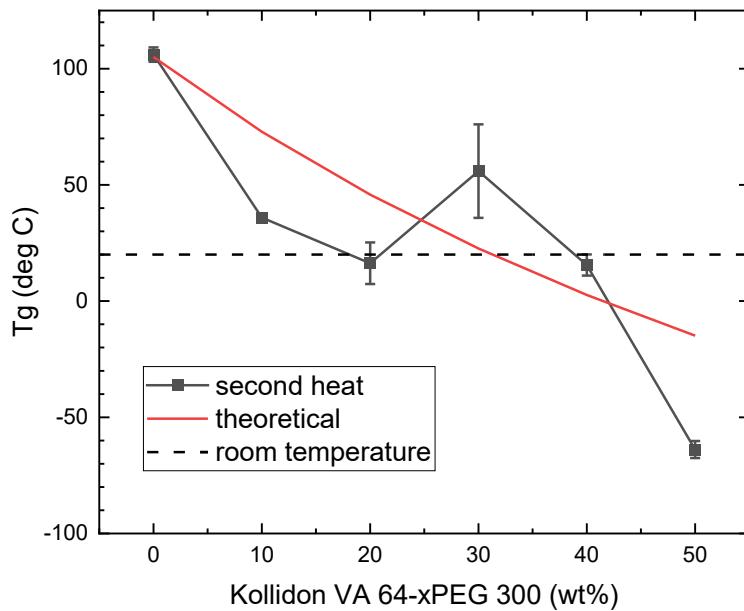


Figure 8. Glass transition temperature of Kollidon VA 64 – PEG300 mixtures as a function of PEG300 content.

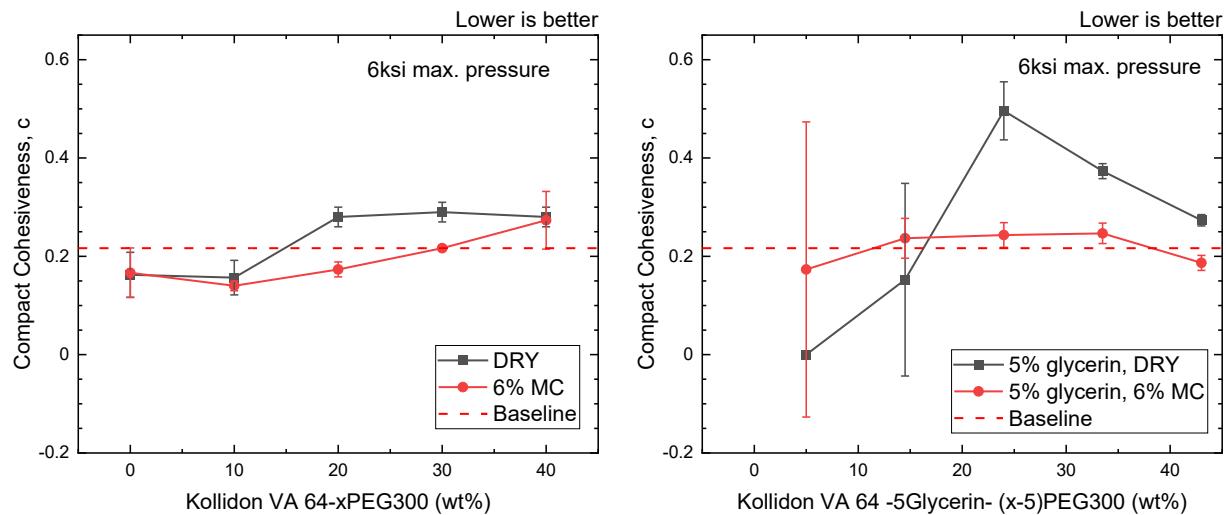
4.2. Kollidon VA Binder System

As expected based on manufacturer data,[1] the Kollidon VA 64 copolymer adsorbed significantly less moisture than the PVP for the same overnight humidity condition (~6 wt.%MC of binder

versus \sim 10.5 wt.%MC of binder). Kollidon VA 64 is a viable alternative to Kollidon 25, as the Kollidon VA 64 compositions performed at least as well as the baseline powders.

The Kollidon VA series showed properties exceeding that of the baseline for compositions in the range of VA64-(0-20 wt.%)PEG300 and VA64-(0-19 wt.%)PEG300-(5 wt.%)Glycerin. In these ranges, property improvements were noted for all properties measured at 40kpsi and humidified except the compactibility coefficient and therefore green density. For these compositions at a maximum pressure of 6kpsi, the properties are about that of the baseline except for the slide coefficient and radial expansion. The compacts should therefore handle about as well as the baseline during uniaxially pressing and better after isostatic. As the dry 0%PEG300 and VA64-10%PEG300 compacts were prone to fracturing at 40kpsi, these compositions should be avoided for production even in their humidified states due to the sensitivity to humidity conditions. Of the binder compositions evaluated, the recommended composition for this powder processing system is 5 wt.% glycerin with a 90:10 ratio of Kollidon VA 64 to PEG300, humidified overnight, as this composition performed the best in terms of cohesiveness and green strength.

This composition still fails to reach the target slide coefficient of at least 0.7 and compact cohesiveness of over 1.0; its properties, seen in Figure 7, are 0.642 and 0.84, respectively. The cohesiveness ratio could possibly be pushed closer to 1 if the in-die friction was reduced. This could be achieved by increasing the lubricant if such could be done without negatively affecting green strength or shrinkage, or by adding a dispersant. Either option should also increase the slide coefficient, as they will reduce friction.



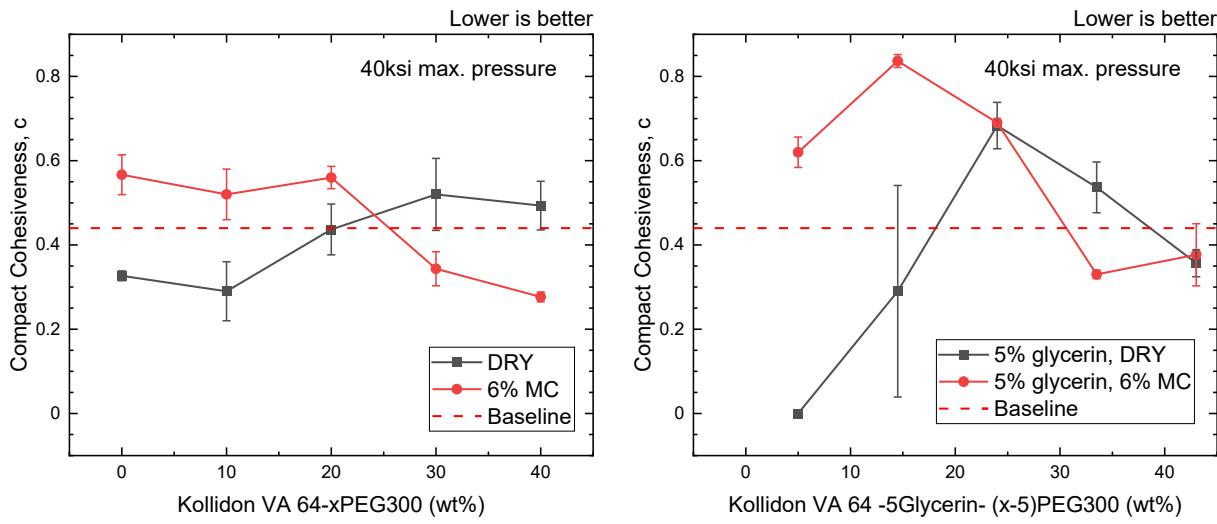


Figure 9. Compact cohesiveness of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin binder.

4.3. Dispersants

The dispersants had no significant effect on most properties of the baseline. All properties were similar between the Baseline and dispersant powders at both pressures and moisture levels. The exceptions are for humidified powders at 40kpsi, where the ejection pressure, stripping pressure, and axial expansion were about 20%, 10%, and 5% lower, respectively, for the DisperBYK powder compared to the baseline. This would suggest reduced in-die friction with the dispersant addition, but there was no significant difference in slide coefficient. The Carbosperse powder had a lower cohesiveness (~30%), axial green strength (~27%), and axial expansion (~14%) than the baseline at 6kpsi, humidity conditioned. There was also variation between the baseline and dispersant powders in the radial and diametral green strength; however, insufficient data points could be collected for these properties to be reliable. The PTC was not able to collect radial crush test data for several samples, as the values were outside the measurement threshold.

As the effect of dispersants on the baseline is not significant for most compaction properties, dispersants could be added to improve other processing steps without detrimental effects on pressing. If one of the dispersants tested is added, it should be the DisperBYK. The DisperBYK powder performed better than the Carbosperse in terms of axial expansion, green density, and ejection and stripping pressure at 40kpsi and axial expansion at 6kpsi.

Unexpectedly, the dried baseline powders and those with dispersants had a greater axial strength and cohesiveness ratio than the same humidified powders at 40kpsi. The humidified baseline powder from the VA64 series and the Dispersant/Alternate Binder series had cohesiveness ratios and axial green strengths within one standard deviation of each other, while the Dispersant/Alternate Binder dry cohesiveness ratio and axial green strength were 0.11 and 216psi greater than the VA64 series, respectively. The PTC was cleaned and calibrated between uses, and all other properties are like the VA64 baseline except the ejection pressure which is about 400psi lower for the Dispersant/Alternate Binder baseline. The lower ejection pressure likely produced less internal stress in the compact, leading to higher axial green strengths, but the cause of the lower ejection pressure is not clear. The difference is not likely due to the difference in the powder batch, as the same lot of

powder was used, but the particle size distribution is shifted slightly to larger particles for the Dispersant/Alternate Binder series.

4.4. Alternate Binder

The compact cohesiveness and axial green strength, shown in Figure 10, of the humidified powders were \sim 63-65% and \sim 53-58% lower for the Aquazol-PEG6000 than the baseline at both pressures. The radial green strength at 40kpsi was 54% higher for the Aquazol-PEG6000 than baseline, however. The main benefit of the Aquazol-PEG6000 is the lower slide coefficient (\sim 8% at 6kpsi) and lower stripping pressure (\sim 35-40%). The Aquazol-PEG6000 powder had less in-die friction but was much weaker than the baseline. The compactibility and out-of-die green density were also higher for the baseline than the Aquazol-PEG6000 at both 6kpsi and 40kpsi.

The low strength of the Aquazol-PEG6000 powder limits its use in this process. This is evident in the delamination fractures observed in the dried Aquazol-PEG6000 powder. The Kollidon 25-PEG300 system is therefore a more viable option than the Aquazol-PEG6000.

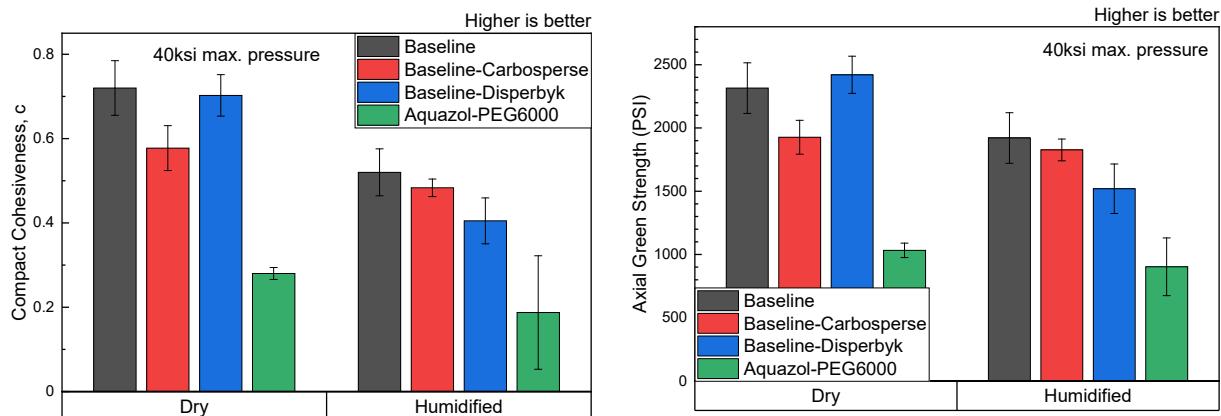


Figure 10. The compact cohesiveness and axial green strength of the Dispersant / Alternate Binder compacts at 40 kpsi max pressure.

5. SUMMARY

This SAND Report provides experimental data for an organic binder system for PZT 95/5 consisting of Kollidon VA 64, PEG300, and glycerin. The study explored the glass transition temperature, powder compaction properties, and green strengths over a wide composition range. The composition Kollidon VA 64 – 20 PEG300 – 5 Glycerin offered the best combination of properties for the compositions studied. When compared to a baseline binder composition of Kollidon 25 – 30 PEG300, this binder offered improved performance. The results showed that this composition offered improvements to density, frictional forces during pressing and ejection, expansion, strength, and compact cohesiveness. Additionally, this study showed that Kollidon VA 64 has lower moisture sensitivity compared to Kollidon 25. This study also compared the baseline binder to an Aquazol 200 – PEG6000 binder system and the effect of two dispersants to the baseline binder. The baseline binder was measured to have better performance compared to the Aquazol 200 based binder, primarily because the Aquazol 200 based binder appeared to be underplasticized in the present study. The DisperBYK-2155 and Carbosperse K-XP22B dispersants did not noticeable alter the compaction properties, suggesting good compatibility with the binder system.

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APPENDIX A. TG DETERMINATION VIA DSC

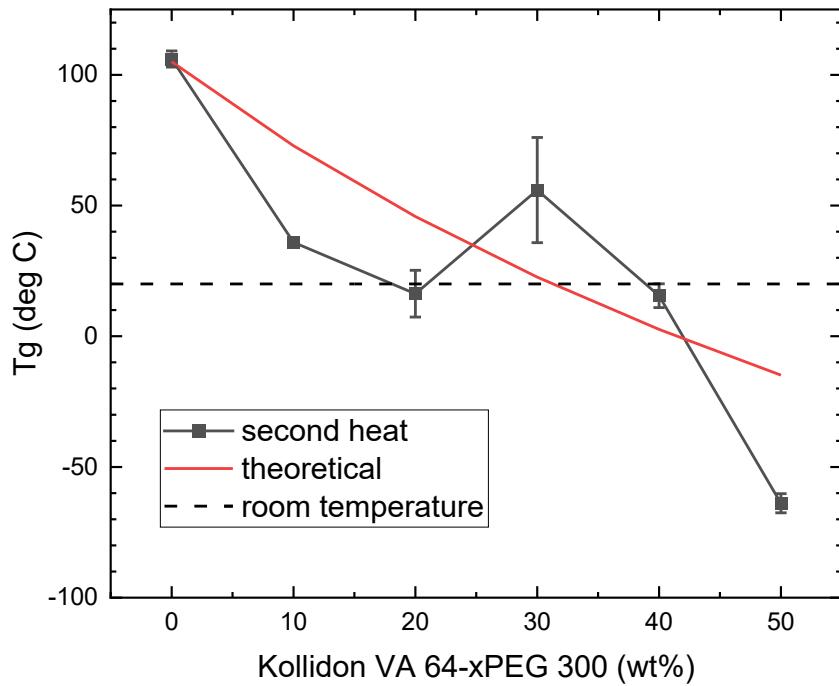


Figure A-1. The T_g of Kollidon VA 64 as a function of plasticizer content is shown above.

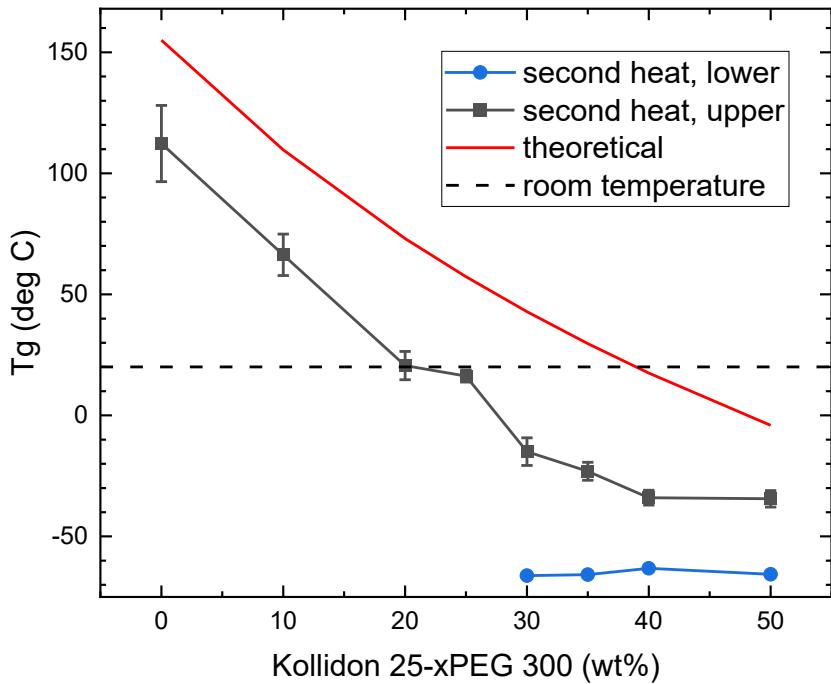


Figure A-2. The T_g of Kollidon 25 as a function of plasticizer content is shown above.

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APPENDIX B. KOLLIDON VA 64 – PEG300 COMPACTION PROPERTIES

Table B-I. Compaction properties of Kollidon VA 64 – xPEG300 powders at 40 kpsi max. pressure, humidified overnight

| Sample ID | Baseline | Baseline+PF | VA64-0%PEG300 | VA64-10%PEG300 | VA64-20%PEG300 | VA64-30%PEG300 | VA64-40%PEG300 |
|---|---------------|---------------|---------------|----------------|----------------|----------------|----------------|
| Moisture Content (wt.% binder) [†] | ~10.8 | ~10.7 | ~5.7 | ~6.4 | ~6.6 | ~6.8 | ~6.8 |
| Compact Cohesiveness, c | 0.44 ± 0.03 | 0.39 ± 0.02 | 0.57 ± 0.05 | 0.52 ± 0.06 | 0.56 ± 0.03 | 0.34 ± 0.04 | 0.28 ± 0.01 |
| Compactibility Coefficient, α | 0.128 ± 0.001 | 0.119 ± 0.000 | 0.117 ± 0.001 | 0.122 ± 0.001 | 0.128 ± 0.001 | 0.134 ± 0.000 | 0.140 ± 0.000 |
| Slide Coefficient, η | 0.610 ± 0.003 | 0.612 ± 0.003 | 0.647 ± 0.002 | 0.650 ± 0.002 | 0.642 ± 0.003 | 0.613 ± 0.006 | 0.603 ± 0.002 |
| Axial Expansion (%) | 3.48 ± 0.05 | 3.65 ± 0.03 | 2.83 ± 0.05 | 2.83 ± 0.04 | 3.15 ± 0.03 | 3.42 ± 0.02 | 3.79 ± 0.01 |
| Radial Expansion (%) | 0.68 ± 0.02 | 0.65 ± 0.01 | 0.53 ± 0.01 | 0.58 ± 0.00 | 0.64 ± 0.02 | 0.64 ± 0.02 | 0.67 ± 0.04 |
| Ejection Pressure (psi) | 2182 ± 27 | 1922 ± 49 | 1591 ± 43 | 1552 ± 6 | 1652 ± 27 | 2108 ± 64 | 3017 ± 102 |
| Stripping Pressure (psi) | 897 ± 7 | 732 ± 16 | 510 ± 17 | 518 ± 9 | 652 ± 31 | 895 ± 35 | 1267 ± 17 |
| Green Density (g/cc) | 4.890 ± 0.008 | 4.724 ± 0.005 | 4.755 ± 0.008 | 4.841 ± 0.007 | 4.925 ± 0.006 | 5.003 ± 0.005 | 5.075 ± 0.003 |
| Axial Green Strength (psi) | 1773 ± 127 | 1569 ± 70 | 2046 ± 182 | 1859 ± 213 | 2068 ± 110 | 1368 ± 141 | 1137 ± 44 |
| Radial Green Strength (psi) | 1561 ± 297 | 1541 ± 188 | 1164 ± 684 | 1265 ± 776 | 2022 ± 171 | 1656 ± 140 | 1726 ± 382 |
| Diametral Green Strength (psi) | 63 ± 10 | 66 ± 36 | 53 ± 16 | 71 ± 20 | 87 ± 36 | 86 ± 7 | 72 ± 27 |

[†]wt.% normalized to binder and plasticizer content

Table B-II. Compaction properties of Kollidon VA 64-xPEG300 powders at 6kpsi max. pressure, humidified overnight

| Sample ID | Baseline | Baseline+PF | VA64-0%PEG300 | VA64-10%PEG300 | VA64-20%PEG300 | VA64-30%PEG300 | VA64-40%PEG300 |
|---------------------------------|---------------|---------------|---------------|----------------|----------------|----------------|----------------|
| Moisture Content (wt.% binder)† | ~10.6 | ~10.9 | ~5.2 | ~5.5 | ~6.4 | ~6.5 | ~6.6 |
| Compact Cohesiveness, c | 0.22 ± 0.03 | 0.27 ± 0.04 | 0.17 ± 0.05 | 0.14 ± 0.01 | 0.17 ± 0.02 | 0.22 ± 0.01 | 0.27 ± 0.06 |
| Compactibility Coefficient, α | 0.115 ± 0.002 | 0.105 ± 0.001 | 0.101 ± 0.000 | 0.106 ± 0.000 | 0.112 ± 0.001 | 0.125 ± 0.000 | 0.130 ± 0.000 |
| Slide Coefficient, η | 0.563 ± 0.012 | 0.563 ± 0.007 | 0.505 ± 0.008 | 0.516 ± 0.010 | 0.493 ± 0.032 | 0.562 ± 0.005 | 0.528 ± 0.009 |
| Axial Expansion (%) | 1.38 ± 0.05 | 1.38 ± 0.03 | 1.01 ± 0.04 | 0.84 ± 0.02 | 0.85 ± 0.01 | 1.26 ± 0.02 | 1.52 ± 0.05 |
| Radial Expansion (%) | 0.24 ± 0.01 | 0.23 ± 0.02 | 0.26 ± 0.01 | 0.26 ± 0.03 | 0.25 ± 0.02 | 0.24 ± 0.00 | 0.27 ± 0.01 |
| Ejection Pressure (psi) | 206 ± 13 | 182 ± 6 | 230 ± 1 | 177 ± 1 | 144 ± 6 | 220 ± 8 | 324 ± 1 |
| Stripping Pressure (psi) | 99 ± 7 | 85 ± 4 | 78 ± 1 | 68 ± 1 | 70 ± 1 | 124 ± 4 | 183 ± 0 |
| Green Density (g/cc) | 4.241 ± 0.023 | 4.096 ± 0.020 | 4.010 ± 0.001 | 4.099 ± 0.006 | 4.183 ± 0.010 | 4.412 ± 0.002 | 4.464 ± 0.001 |
| Axial Green Strength (psi) | 146 ± 19 | 185 ± 22 | 120 ± 34 | 100 ± 7 | 129 ± 11 | 149 ± 4 | 200 ± 44 |
| Radial Green Strength (psi) | ND‡ | ND‡ | ND‡ | ND‡ | ND‡ | ND‡ | ND‡ |
| Diametral Green Strength (psi) | 21* | ND‡ | 15* | ND‡ | ND‡ | ND‡ | 25* |

†wt.% normalized to binder and plasticizer content

‡strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table B-III. Compaction properties of Kollidon VA 64 – xPEG300 powders at 40 kpsi max. pressure, dried

| Sample ID | Baseline | Baseline+PF | VA64-0%PEG300 | VA64-10%PEG300 | VA64-20%PEG300 | VA64-30%PEG300 | VA64-40%PEG300 |
|---|---------------|---------------|---------------|----------------|----------------|----------------|----------------|
| Moisture Content (wt.% binder) [†] | ~0.3 | ~0.3 | ~0.3 | ~0.5 | ~0.0 | ~0.0 | ~0.0 |
| Compact Cohesiveness, c | 0.48 ± 0.05 | 0.40 ± 0.02 | 0.33 ± 0.01 | 0.29 ± 0.07 | 0.44 ± 0.06 | 0.52 ± 0.09 | 0.49 ± 0.06 |
| Compactibility Coefficient, α | 0.124 ± 0.001 | 0.115 ± 0.001 | 0.117 ± 0.000 | 0.120 ± 0.000 | 0.125 ± 0.001 | 0.131 ± 0.001 | 0.136 ± 0.001 |
| Slide Coefficient, η | 0.643 ± 0.004 | 0.641 ± 0.003 | 0.656 ± 0.014 | 0.650 ± 0.007 | 0.652 ± 0.007 | 0.650 ± 0.008 | 0.648 ± 0.007 |
| Axial Expansion (%) | 3.42 ± 0.01 | 3.49 ± 0.06 | 3.24 ± 0.31 | 3.03 ± 0.06 | 3.29 ± 0.01 | 3.51 ± 0.07 | 3.49 ± 0.14 |
| Radial Expansion (%) | 0.70 ± 0.02 | 0.62 ± 0.01 | 0.60 ± 0.05 | 0.60 ± 0.02 | 0.65 ± 0.01 | 0.70 ± 0.02 | 0.69 ± 0.02 |
| Ejection Pressure (psi) | 1748 ± 14 | 1671 ± 12 | 1587 ± 4 | 1639 ± 38 | 1576 ± 18 | 1604 ± 42 | 1892 ± 71 |
| Stripping Pressure (psi) | 602 ± 16 | 538 ± 8 | 512 ± 6 | 514 ± 10 | 525 ± 25 | 605 ± 45 | 770 ± 51 |
| Green Density (g/cc) | 4.831 ± 0.007 | 4.676 ± 0.006 | 4.718 ± 0.019 | 4.788 ± 0.007 | 4.859 ± 0.017 | 4.952 ± 0.014 | 5.033 ± 0.008 |
| Axial Green Strength (psi) | 1737 ± 162 | 1476 ± 76 | 1153 ± 66 | 1030 ± 234 | 1561 ± 246 | 1866 ± 267 | 1792 ± 189 |
| Radial Green Strength (psi) | 1766 ± 241 | 2043 ± 483 | 943 ± 832 | 1403 ± 116 | 1561 ± 279 | 1365 ± 119 | 2046 ± 50 |
| Diametral Green Strength (psi) | 67 ± 8 | 93 ± 16 | 57 ± 30 | 49 ± 17 | 52 ± 6 | 73 ± 14 | 82 ± 27 |

[†]wt.% normalized to binder and plasticizer content

Table B-IV. Compaction properties of Kollidon VA 64 – xPEG300 powders at 6 kpsi max. pressure, dried.

| Sample ID | Baseline | Baseline+PF | VA64-0%PEG300 | VA64-10%PEG300 | VA64-20%PEG300 | VA64-30%PEG300 | VA64-40%PEG300 |
|---|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|----------------|
| Moisture Content (wt.% binder) [†] | ~0.3 | ~0.5 | ~0.0 | ~0.3 | ~0.0 | ~0.0 | ~0.0 |
| Compact Cohesiveness, c | 0.25 ± 0.02 | 0.18 ± 0.02 | 0.16 ± 0.05 | 0.16 ± 0.04 | 0.28 ± 0.02 | 0.29 ± 0.02 | 0.28 ± 0.02 |
| Compactibility Coefficient, α | 0.106 ± 0.003 | 0.102 ± 0.004 | 0.102 ± 0.000 | 0.105 ± 0.000 | 0.107 ± 0.001 | 0.118 ± 0.001 | 0.127 ± 0.001 |
| Slide Coefficient, η | 0.581 ± 0.024 | 0.531 ± 0.046 | 0.608 ± 0.004 | 0.613 ± 0.007 | 0.574 ± 0.009 | 0.586 ± 0.005 | 0.565 ± 0.006 |
| Axial Expansion (%) | 1.23 ± 0.05 | 1.56 ± 0.16 | 1.10 ± 0.16 | 1.12 ± 0.20 | 0.99 ± 0.06 | 1.07 ± 0.06 | 1.23 ± 0.04 |
| Radial Expansion (%) | 0.25 ± 0.02 | 0.29 ± 0.04 | 0.24 ± 0.01 | 0.23 ± 0.02 | 0.25 ± 0.01 | 0.23 ± 0.01 | 0.26 ± 0.02 |
| Ejection Pressure (psi) | 174 ± 03 | 184 ± 3 | 216 ± 6 | 192 ± 3 | 170 ± 2 | 158 ± 4 | 209 ± 10 |
| Stripping Pressure (psi) | 72 ± 5 | 83 ± 5 | 79 ± 1 | 70 ± 1 | 67 ± 1 | 72 ± 3 | 106 ± 9 |
| Green Density (g/cc) | 4.124 ± 0.051 | 4.030 ± 0.062 | 4.069 ± 0.007 | 4.121 ± 0.012 | 4.146 ± 0.013 | 4.320 ± 0.025 | 4.436 ± 0.010 |
| Axial Green Strength (psi) | 163 ± 20 | 133 ± 14 | 99 ± 28 | 92 ± 20 | 187 ± 17 | 187 ± 11 | 191 ± 15 |
| Radial Green Strength (psi) | ND [‡] | 1267* |
| Diametral Green Strength (psi) | 19 ± 7 | 27* | ND [‡] | 21 ± 0 | 21 ± 1 | ND [‡] | 28 ± 1 |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table B-V. Compaction properties of Kollidon VA 64 – 5 Glycerin – (x-5)PEG300 powders at 40 kpsi max. pressure, humidified overnight.

| Sample ID | Baseline | VA64-0%PEG300-5%Glycerin | VA64-10%PEG300-5%Glycerin | VA64-20%PEG300-5%Glycerin | VA64-30%PEG300-5%Glycerin | VA64-40%PEG300-5%Glycerin |
|---|---------------|--------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Moisture Content (wt.% binder) [†] | ~10.8 | ~5.9 | ~6.6 | ~6.8 | ~6.8 | ~6.2 |
| Compact Cohesiveness, <i>c</i> | 0.44 ± 0.03 | 0.62 ± 0.04 | 0.84 ± 0.02 | 0.69 ± 0.01 | 0.33 ± 0.01 | 0.38 ± 0.07 |
| Compactibility Coefficient, <i>α</i> | 0.128 ± 0.001 | 0.116 ± 0.000 | 0.122 ± 0.000 | 0.126 ± 0.000 | 0.130 ± 0.000 | 0.134 ± 0.000 |
| Slide Coefficient, <i>η</i> | 0.610 ± 0.003 | 0.641 ± 0.001 | 0.642 ± 0.002 | 0.649 ± 0.003 | 0.614 ± 0.001 | 0.618 ± 0.001 |
| Axial Expansion (%) | 3.48 ± 0.05 | 2.82 ± 0.04 | 2.95 ± 0.05 | 3.42 ± 0.03 | 3.56 ± 0.04 | 3.45 ± 0.03 |
| Radial Expansion (%) | 0.68 ± 0.02 | 0.58 ± 0.01 | 0.62 ± 0.01 | 0.71 ± 0.01 | 0.67 ± 0.01 | 0.68 ± 0.01 |
| Ejection Pressure (psi) | 2182 ± 27 | 1867 ± 8 | 1811 ± 9 | 1601 ± 11 | 2177 ± 27 | 2585 ± 49 |
| Stripping Pressure (psi) | 897 ± 7 | 551 ± 4 | 603 ± 3 | 697 ± 5 | 1023 ± 11 | 1160 ± 15 |
| Green Density (g/cc) | 4.890 ± 0.008 | 4.730 ± 0.005 | 4.824 ± 0.005 | 4.864 ± 0.002 | 4.928 ± 0.001 | 4.999 ± 0.000 |
| Axial Green Strength (psi) | 1773 ± 127 | 2271 ± 120 | 3078 ± 71 | 2489 ± 13 | 1305 ± 45 | 1486 ± 284 |
| Radial Green Strength (psi) | 1561 ± 297 | 2339 ± 284 | 2296 ± 311 | 1664 ± 333 | 1347 ± 102 | 2026 ± 693 |
| Diametral Green Strength (psi) | 63 ± 10 | 96 ± 42 | 131 ± 31 | 118 ± 39 | 53 ± 4 | 61 ± 10 |

[†]wt.% normalized to binder and plasticizer content

Table B-VI. Compaction properties of Kollidon VA 64 – 5 Glycerin – (x-5)PEG300 powders at 6kpsi max. pressure, humidified overnight

| Sample ID | Baseline | VA64-0%PEG300-5%Glycerin | VA64-10%PEG300-5%Glycerin | VA64-20%PEG300-5%Glycerin | VA64-30%PEG300-5%Glycerin | VA64-40%PEG300-5%Glycerin |
|---|-----------------|--------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Moisture Content (wt.% binder) [†] | ~10.6 | ~5.9 | ~6.4 | ~6.2 | ~6.6 | ~6.2 |
| Compact Cohesiveness, c | 0.22 ± 0.03 | 0.17 ± 0.30 | 0.24 ± 0.04 | 0.24 ± 0.03 | 0.25 ± 0.02 | 0.19 ± 0.02 |
| Compactibility Coefficient, α | 0.115 ± 0.002 | 0.089 ± 0.000 | 0.098 ± 0.001 | 0.105 ± 0.000 | 0.118 ± 0.001 | 0.124 ± 0.000 |
| Slide Coefficient, η | 0.563 ± 0.012 | 0.552 ± 0.013 | 0.527 ± 0.017 | 0.480 ± 0.025 | 0.471 ± 0.020 | 0.463 ± 0.015 |
| Axial Expansion (%) | 1.38 ± 0.05 | 0.97 ± 0.04 | 0.88 ± 0.02 | 0.91 ± 0.07 | 1.13 ± 0.06 | 1.35 ± 0.03 |
| Radial Expansion (%) | 0.24 ± 0.01 | 0.29 ± 0.00 | 0.29 ± 0.00 | 0.28 ± 0.01 | 0.29 ± 0.02 | 0.30 ± 0.01 |
| Ejection Pressure (psi) | 206 ± 13 | 273 ± 5 | 187 ± 3 | 159 ± 3 | 208 ± 7 | 299 ± 6 |
| Stripping Pressure (psi) | 99 ± 7 | 78 ± 1 | 70 ± 1 | 91 ± 2 | 130 ± 4 | 179 ± 5 |
| Green Density (g/cc) | 4.241 ± 0.023 | 3.831 ± 0.003 | 3.967 ± 0.005 | 4.074 ± 0.011 | 4.257 ± 0.003 | 4.343 ± 0.003 |
| Axial Green Strength (psi) | 146 ± 19 | 333* | 163 ± 23 | 184 ± 12 | 195 ± 15 | 151 ± 12 |
| Radial Green Strength (psi) | ND [‡] | 1200* | ND [‡] | ND [‡] | 907* | ND [‡] |
| Diametral Green Strength (psi) | 21* | 27* | 20* | ND [‡] | 23* | 18* |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table B-VII. Compaction properties of Kollidon VA 64 – 5 Glycerin – (x-5) PEG300 powders at 40 kpsi max. pressure, dried

| Sample ID | Baseline | VA64-0%PEG300-5%Glycerin | VA64-10%PEG300-5%Glycerin | VA64-20%PEG300-5%Glycerin | VA64-30%PEG300-5%Glycerin | VA64-40%PEG300-5%Glycerin |
|---|---------------|--------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Moisture Content (wt.% binder) [†] | ~0.3 | ~1.0 | ~0.3 | ~0.0 | ~0.0 | ~0.0 |
| Compact Cohesiveness, c | 0.48 ± 0.05 | 0.00 ± 0.00 | 0.29 ± 0.25 | 0.68 ± 0.06 | 0.54 ± 0.06 | 0.36 ± 0.03 |
| Compactibility Coefficient, α | 0.124 ± 0.001 | 0.113 ± 0.000 | 0.118 ± 0.001 | 0.121 ± 0.000 | 0.126 ± 0.000 | 0.133 ± 0.001 |
| Slide Coefficient, η | 0.643 ± 0.004 | 0.684 ± 0.009 | 0.649 ± 0.004 | 0.677 ± 0.003 | 0.671 ± 0.004 | 0.654 ± 0.019 |
| Axial Expansion (%) | 3.42 ± 0.01 | 3.56 ± 0.12 | 3.08 ± 0.12 | 3.12 ± 0.02 | 3.54 ± 0.03 | 3.63 ± 0.28 |
| Radial Expansion (%) | 0.70 ± 0.02 | 0.65 ± 0.01 | 0.65 ± 0.02 | 0.66 ± 0.01 | 0.70 ± 0.01 | 0.68 ± 0.00 |
| Ejection Pressure (psi) | 1748 ± 14 | 1591 ± 5 | 1695 ± 57 | 1376 ± 17 | 1449 ± 9 | 1713 ± 197 |
| Stripping Pressure (psi) | 602 ± 16 | 486 ± 2 | 506 ± 10 | 466 ± 4 | 530 ± 8 | 733 ± 142 |
| Green Density (g/cc) | 4.831 ± 0.007 | 4.646 ± 0.006 | 4.746 ± 0.017 | 4.804 ± 0.006 | 4.869 ± 0.002 | 4.972 ± 0.011 |
| Axial Green Strength (psi) | 1737 ± 162 | ND [‡] | 1558 ± 1 | 2256 ± 166 | 1802 ± 189 | 1270 ± 67 |
| Radial Green Strength (psi) | 1766 ± 241 | ND [‡] | 1663* | 1255 ± 674 | 1825 ± 480 | 1522 ± 579 |
| Diametral Green Strength (psi) | 67 ± 8 | ND [‡] | 45* | 55 ± 24 | 84 ± 8 | 71 ± 26 |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table B-VIII. Compaction properties of Kollidon VA 64 – 5 Glycerin – (x-5) PEG300 powders at 6kpsi max. pressure, dried

| Sample ID | Baseline | VA64-0%PEG300-5%Glycerin | VA64-10%PEG300-5%Glycerin | VA64-20%PEG300-5%Glycerin | VA64-30%PEG300-5%Glycerin | VA64-40%PEG300-5%Glycerin |
|---|-----------------|--------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Moisture Content (wt.% binder) [†] | ~0.3 | ~0.0 | ~0.5 | ~0.3 | ~0.0 | ~0.0 |
| Compact Cohesiveness, c | 0.25 ± 0.02 | 0.00 ± 0.00 | 0.15 ± 0.20 | 0.50 ± 0.06 | 0.37 ± 0.02 | 0.27 ± 0.01 |
| Compactibility Coefficient, α | 0.106 ± 0.003 | 0.088 ± 0.001 | 0.094 ± 0.002 | 0.098 ± 0.002 | 0.114 ± 0.002 | 0.123 ± 0.002 |
| Slide Coefficient, η | 0.581 ± 0.024 | 0.542 ± 0.025 | 0.554 ± 0.036 | 0.617 ± 0.007 | 0.577 ± 0.023 | 0.540 ± 0.022 |
| Axial Expansion (%) | 1.23 ± 0.05 | 1.17 ± 0.08 | 1.34 ± 0.07 | 1.34 ± 0.14 | 1.31 ± 0.09 | 1.47 ± 0.03 |
| Radial Expansion (%) | 0.25 ± 0.02 | 0.28 ± 0.01 | 0.27 ± 0.03 | 0.29 ± 0.01 | 0.28 ± 0.01 | 0.28 ± 0.02 |
| Ejection Pressure (psi) | 174 ± 3 | 269 ± 14 | 209 ± 19 | 193 ± 6 | 188 ± 1 | 222 ± 10 |
| Stripping Pressure (psi) | 72 ± 5 | 80 ± 5 | 71 ± 11 | 83 ± 6 | 098 ± 7 | 129 ± 13 |
| Green Density (g/cc) | 4.124 ± 0.051 | 3.809 ± 0.008 | 3.913 ± 0.051 | 4.000 ± 0.037 | 4.246 ± 0.027 | 4.368 ± 0.022 |
| Axial Green Strength (psi) | 163 ± 20 | ND [‡] | 193 ± 107 | 296 ± 39 | 249 ± 23 | 199 ± 11 |
| Radial Green Strength (psi) | ND [‡] | ND [‡] | ND [‡] | ND [‡] | 797 ± 593 | 894* |
| Diametral Green Strength (psi) | 19 ± 7 | ND [‡] | ND [‡] | 22 ± 12 | 31 ± 4 | 31 ± 5 |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

APPENDIX C. KOLLIDON VA 64 – PEG300 COMPACTION PROPERTY GRAPHS

The Baseline in all graphs is the baseline powder, humidified, at either 6kpsi or 40kpsi.

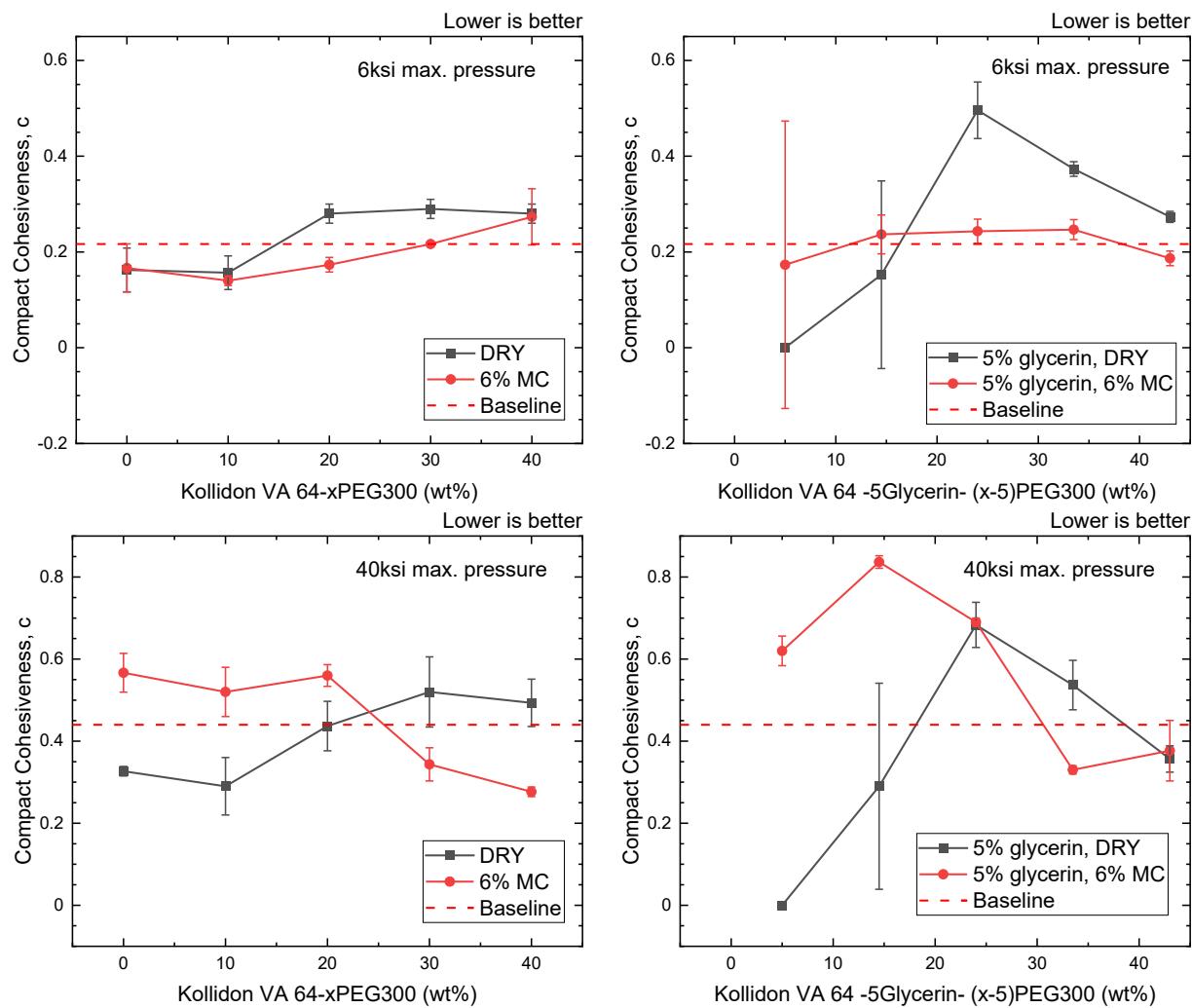


Figure C-1. Compact cohesiveness of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

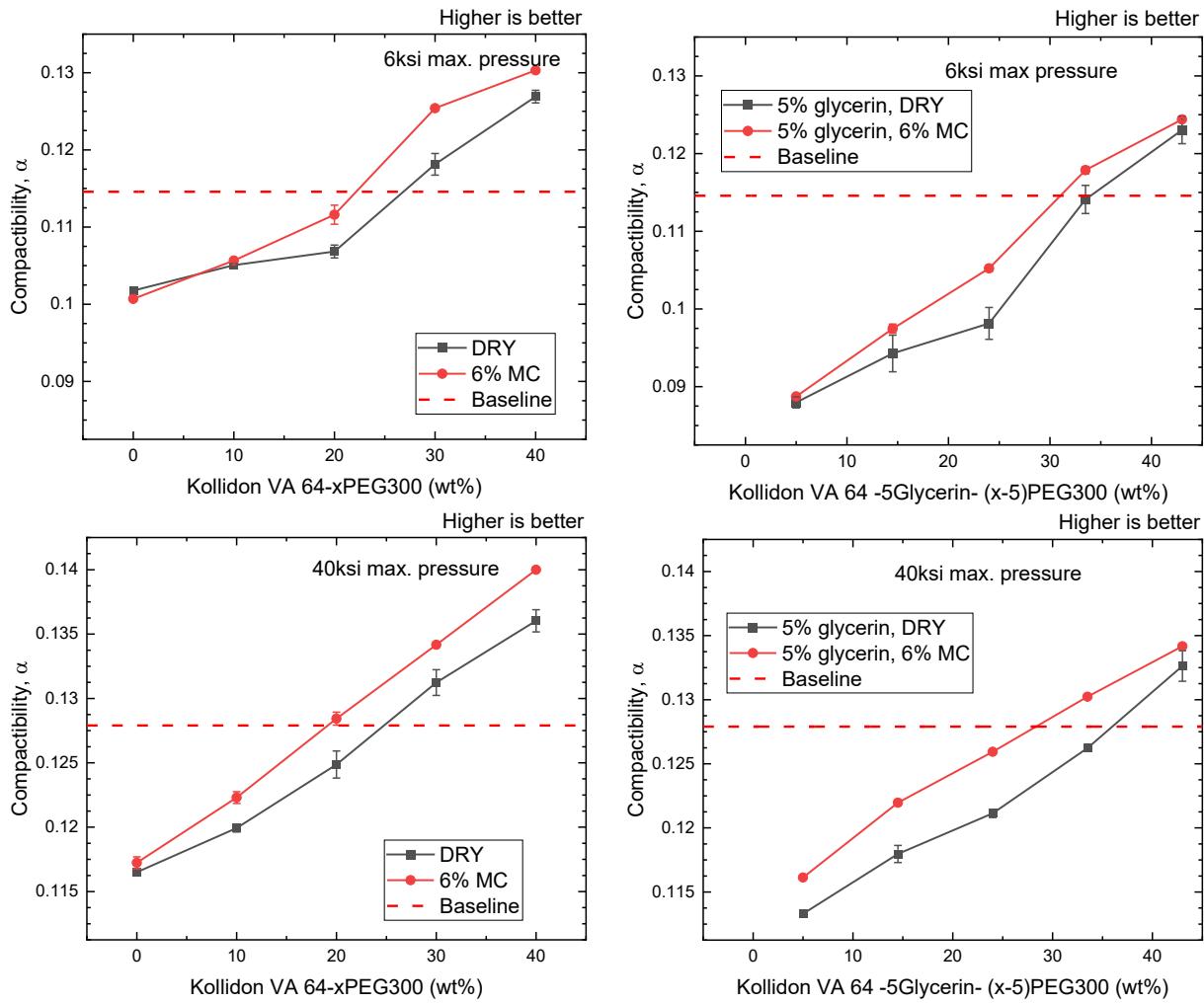


Figure C-2. Compactibility coefficient of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

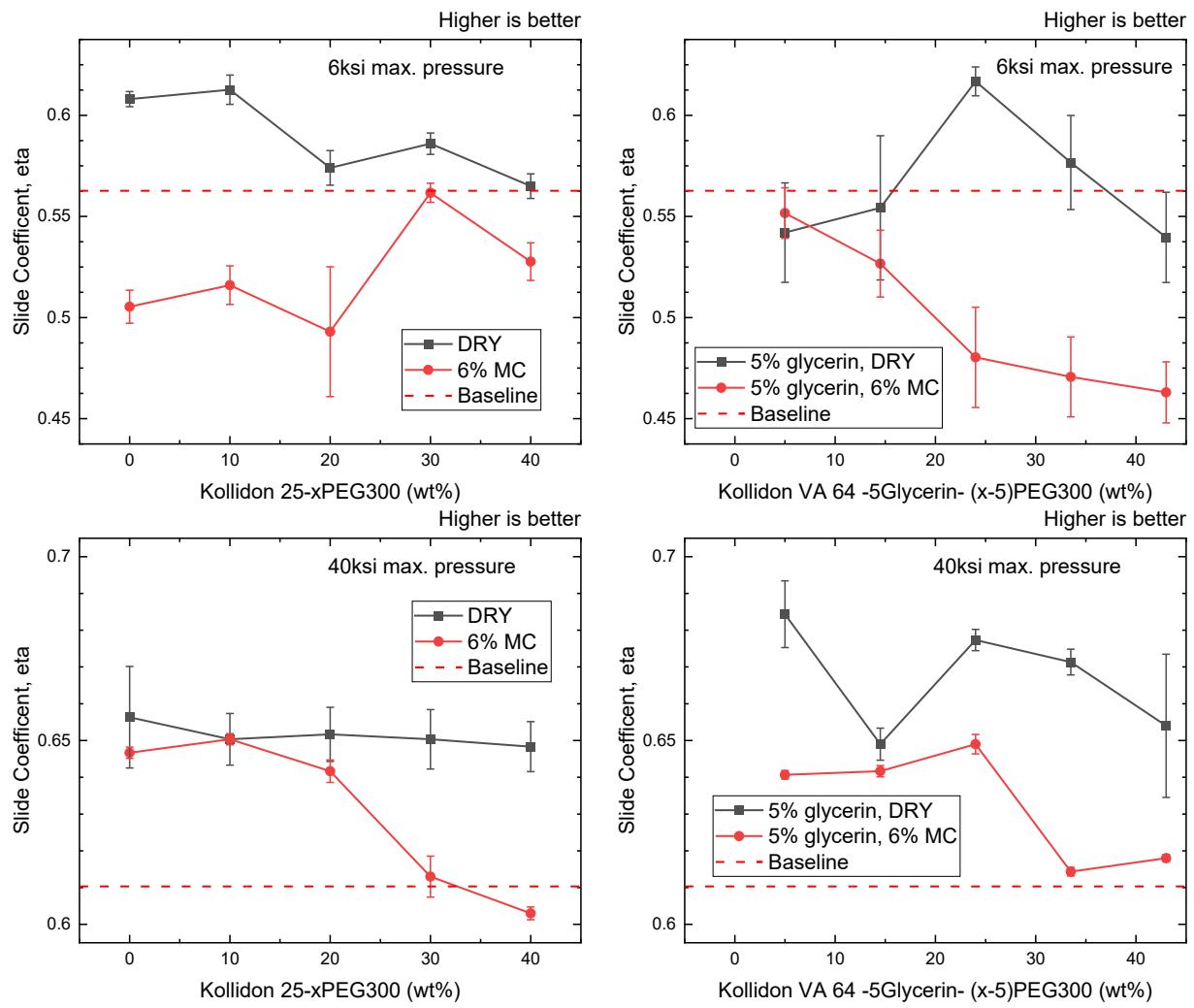


Figure C-3. Slide coefficient of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin – based binders.

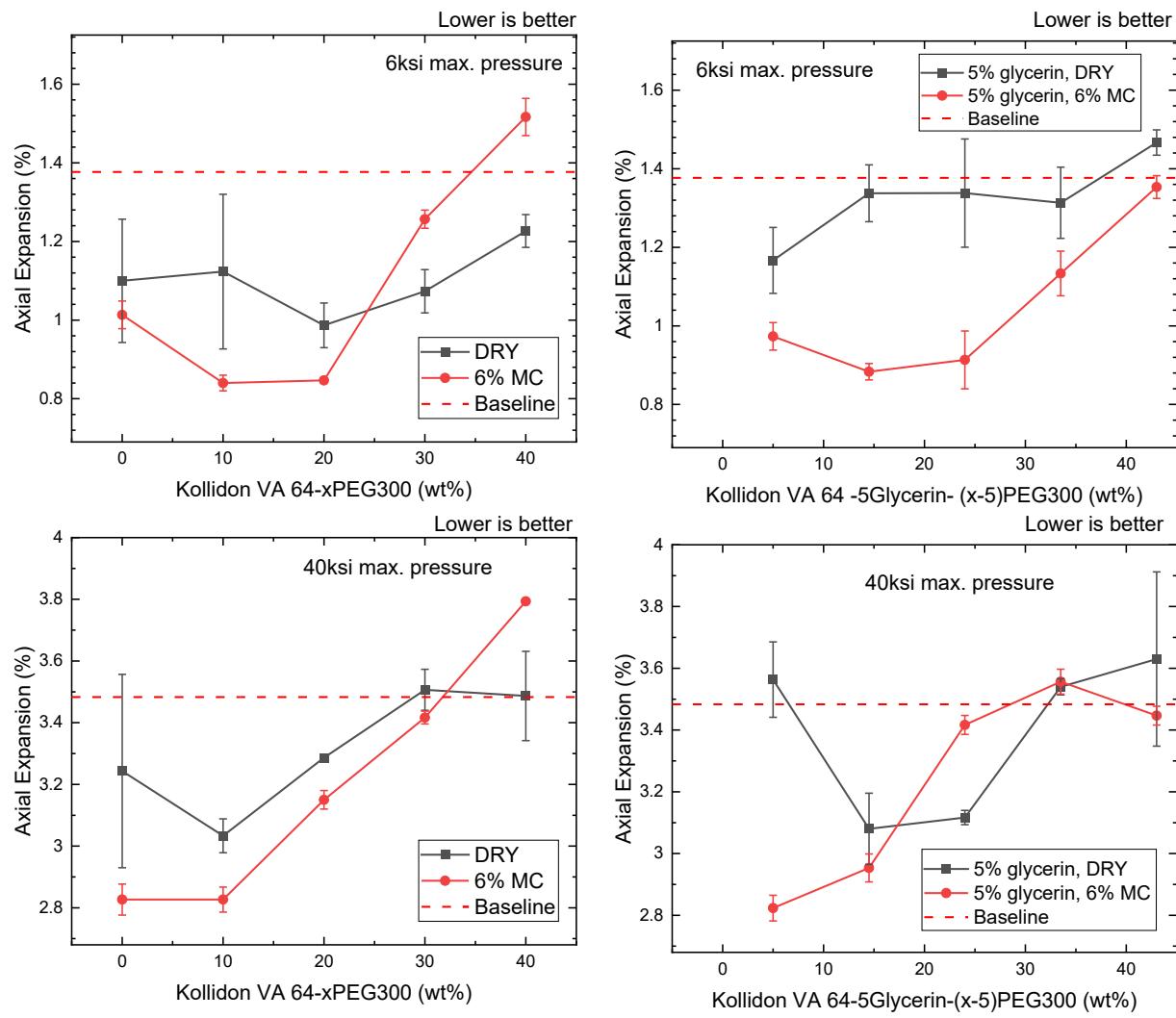
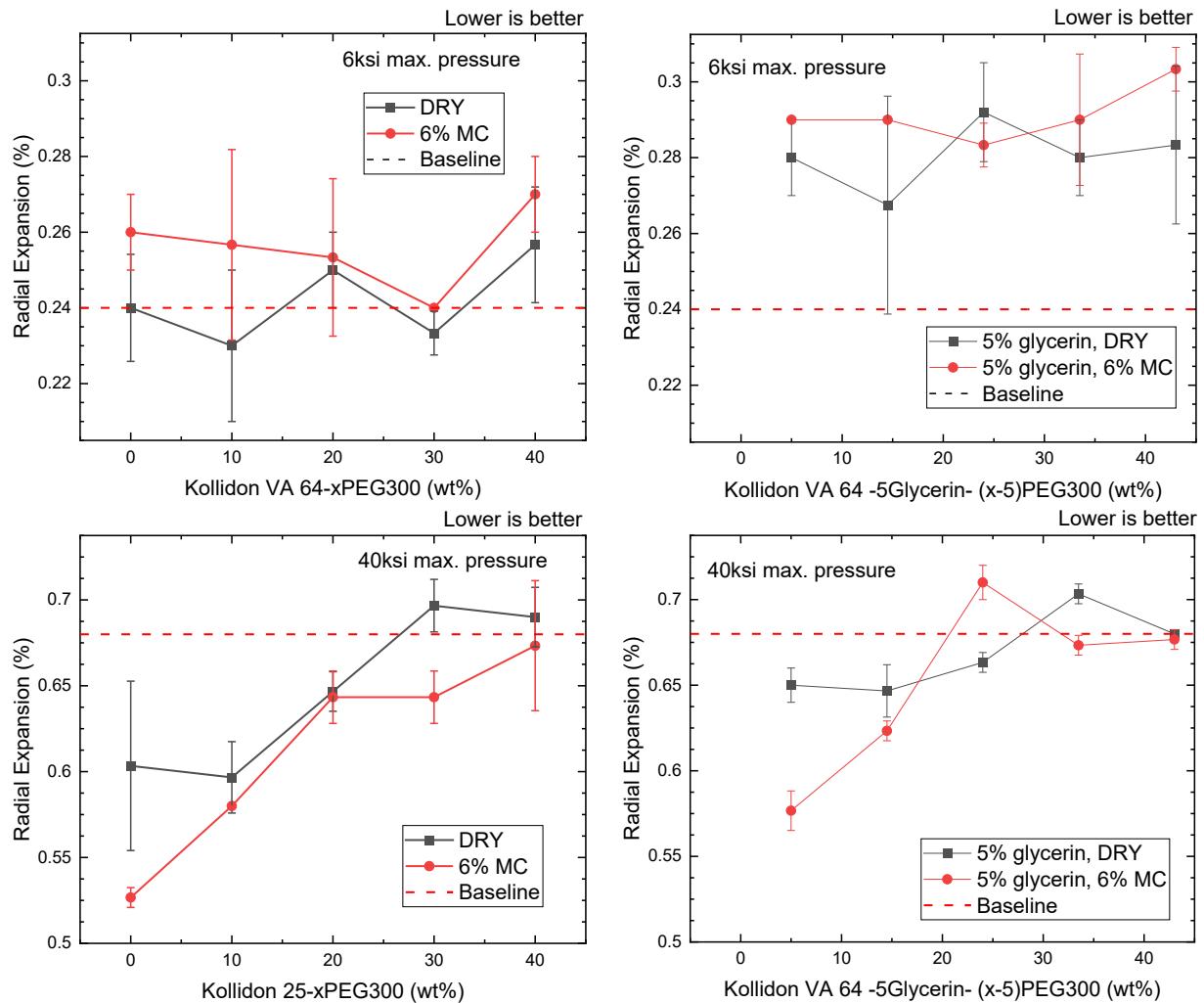


Figure C-4. Axial expansion of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin – based binders.



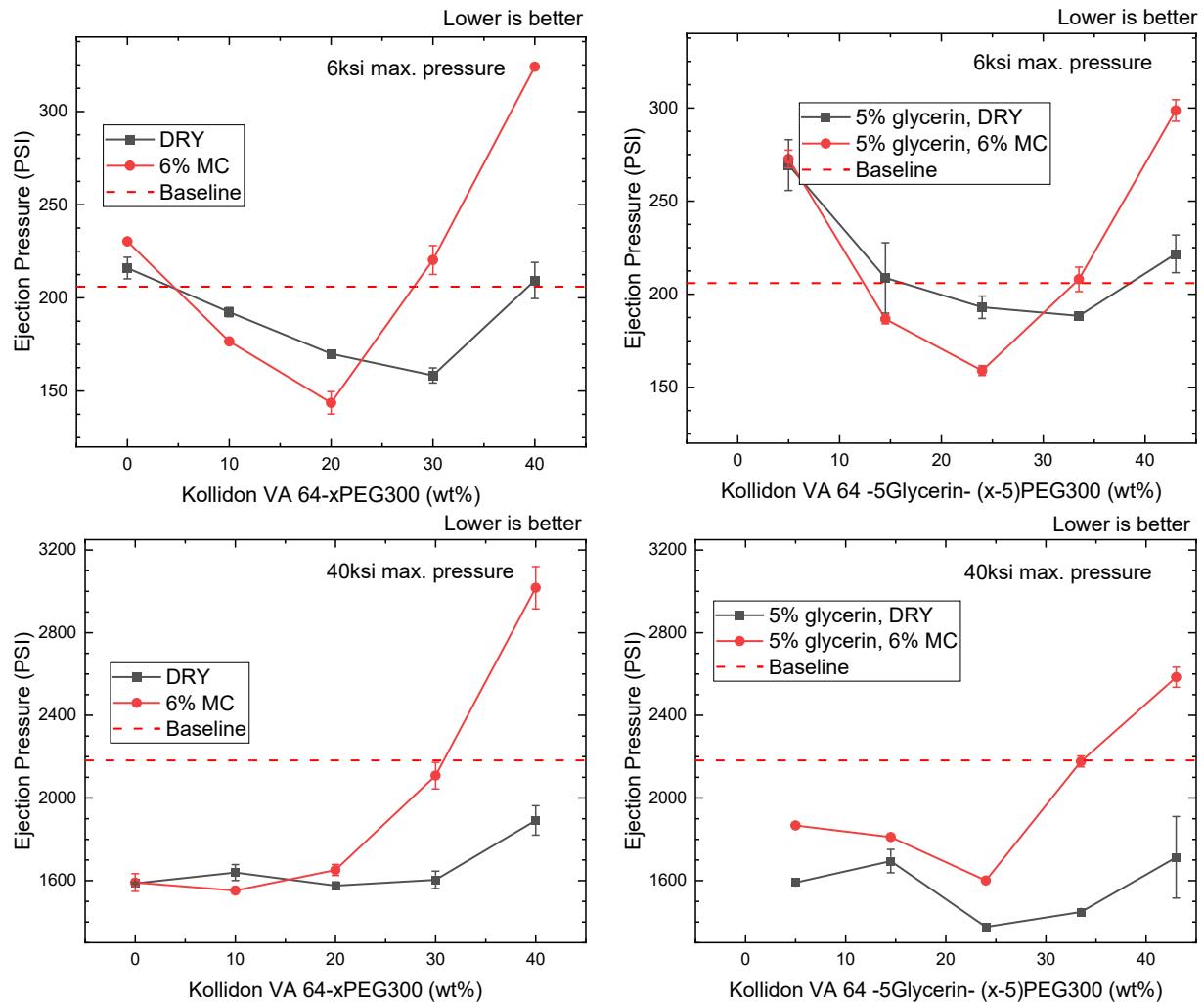


Figure C-6. Ejection pressure of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin – based binders.

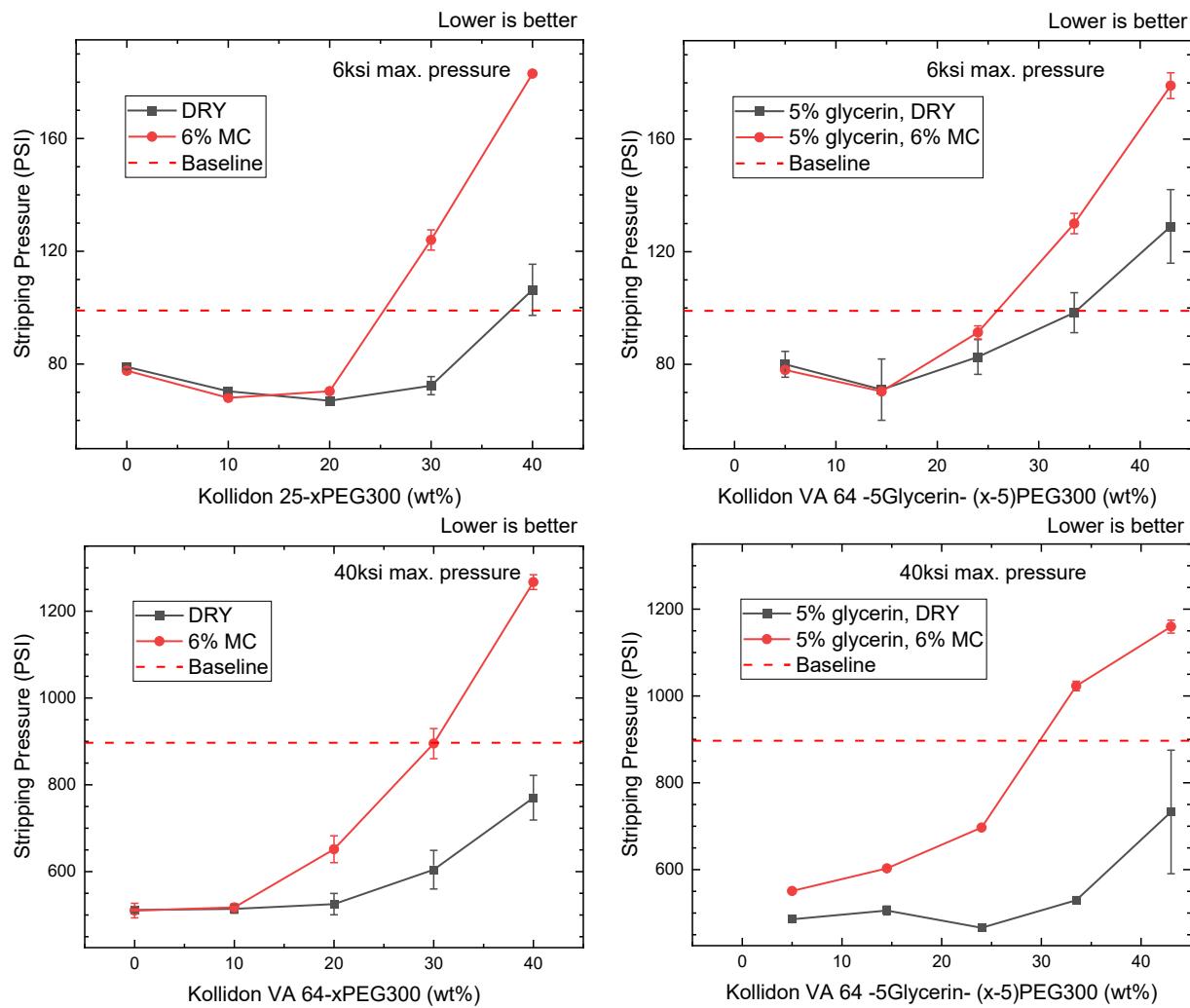


Figure C-7. Stripping pressure of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

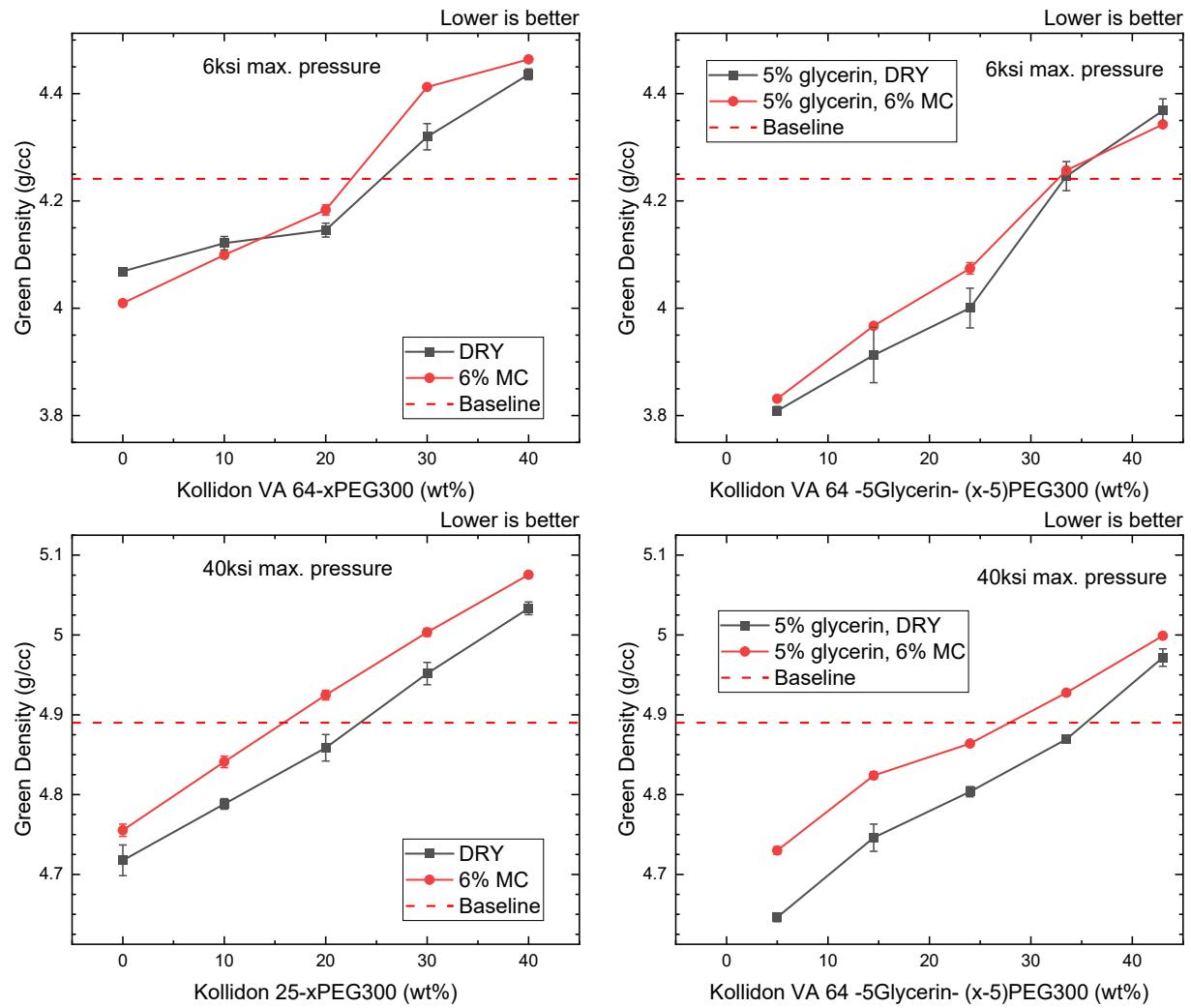


Figure C-8. Out-of-die green density of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

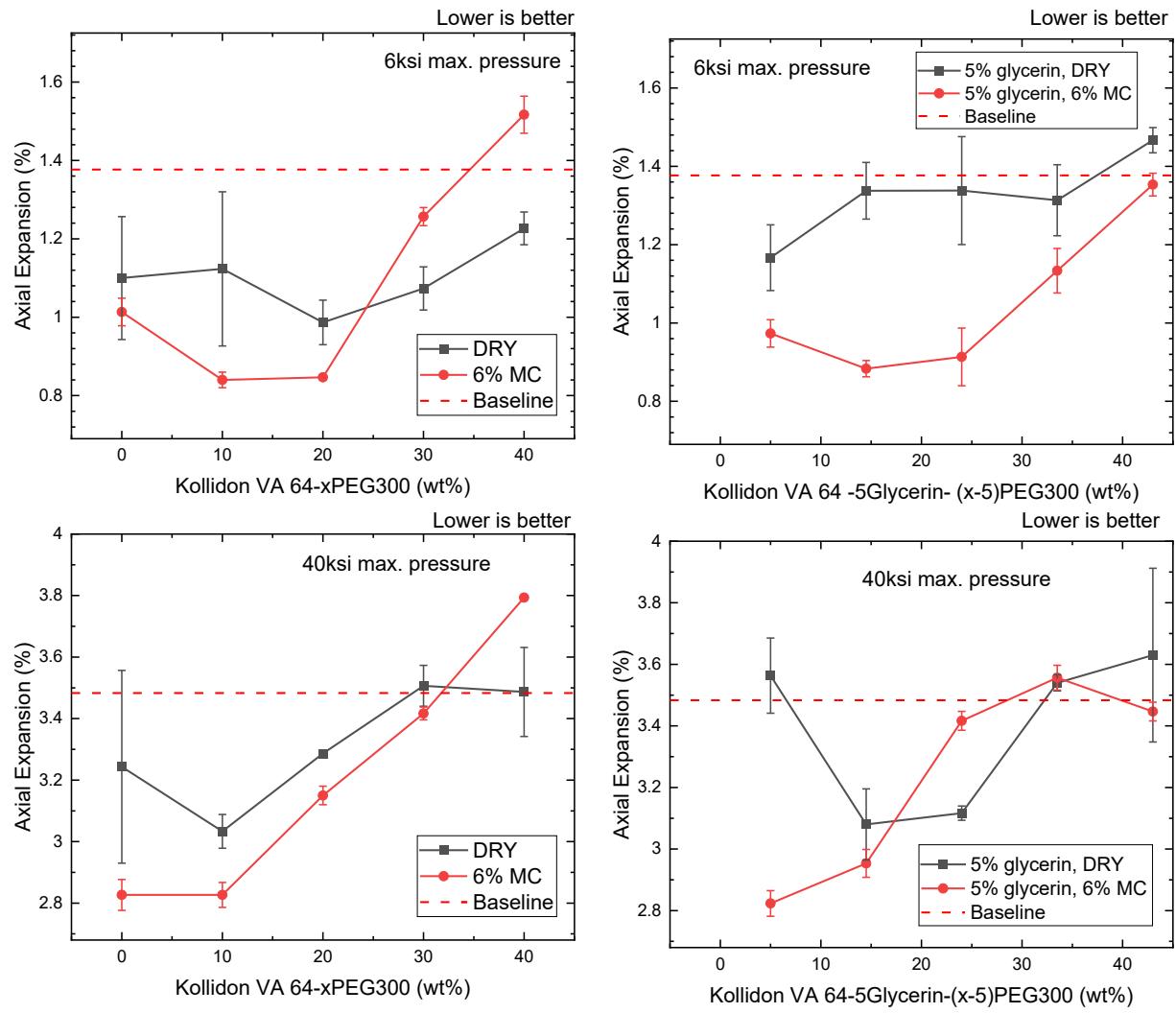


Figure C-9. Axial green of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

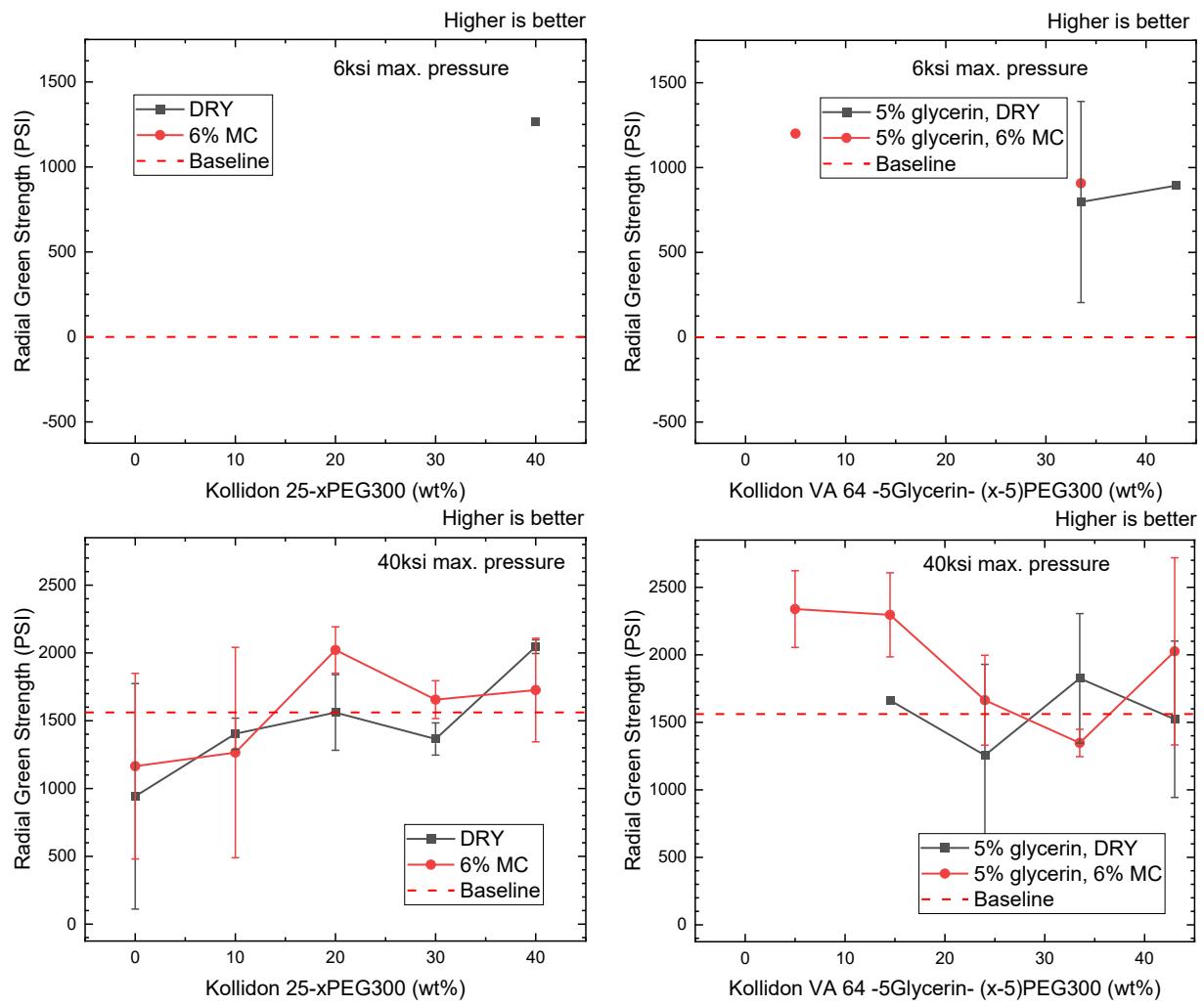


Figure C-10. Radial green strength of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

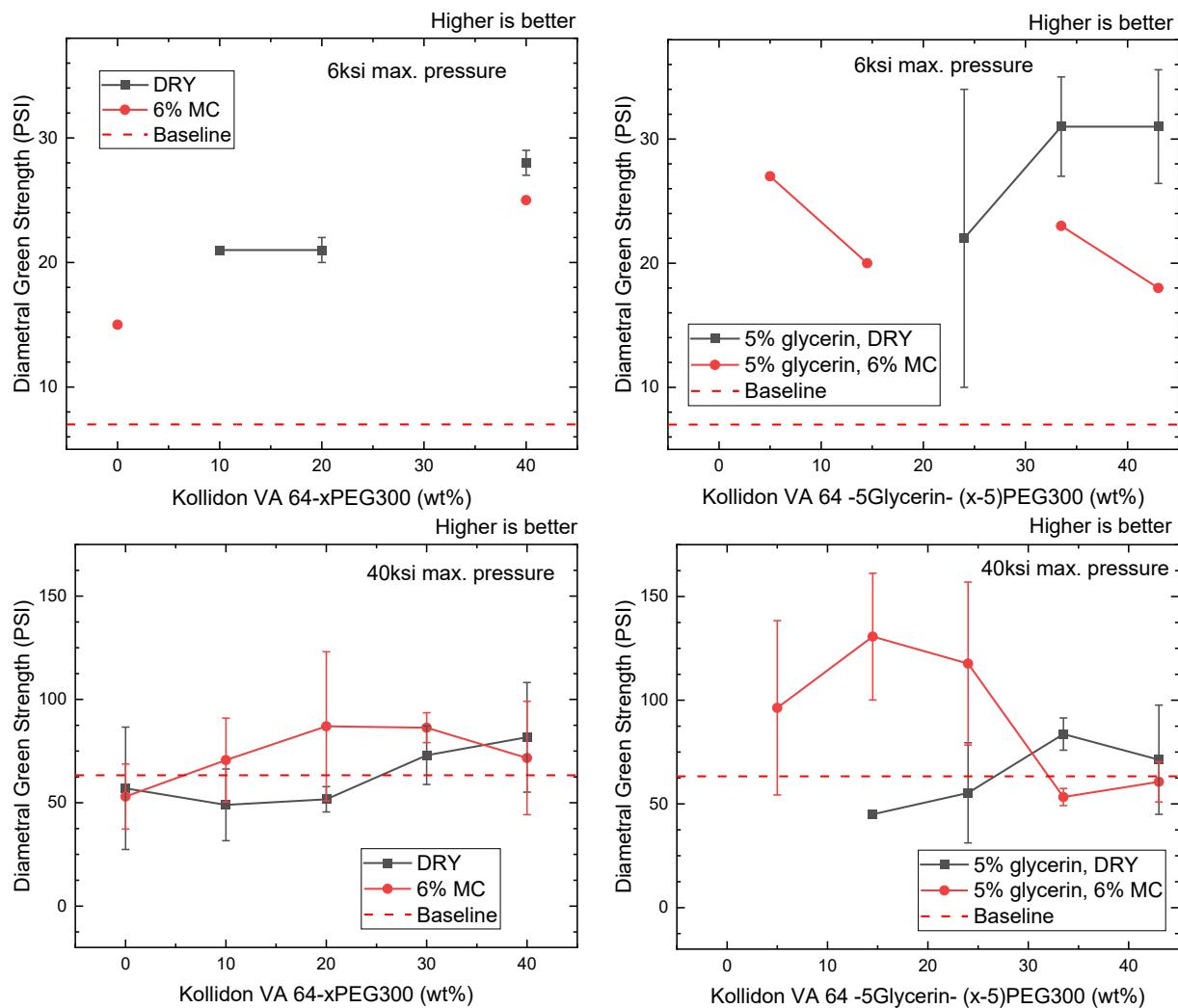


Figure C-11. Diametral green strength of the PZT 95/5 compacts with Kollidon VA 64 – PEG300 – glycerin -based binders.

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APPENDIX D. DISPERSANT/ALTERNATE BINDER COMPACTION PROPERTIES

Table D-I. Compaction Properties of the Dispersant / Alternate binder compacts at 6kpsi max pressure, humidified overnight

| Sample ID | Baseline | Baseline-Carbosperse | Baseline-DisperBYK | Aquazol-PEG6000 |
|---|---------------|----------------------|--------------------|-----------------|
| Moisture Content (wt.% binder) [†] | ~9.7 | ~8.0 | ~9.0 | ~4.3 |
| Compact Cohesiveness, c | 0.31 ± 0.04 | 0.21 ± 0.01 | 0.24 ± 0.03 | 0.11 ± 0.08 |
| Compactibility Coefficient, α | 0.128 ± 0.000 | 0.128 ± 0.000 | 0.132 ± 0.000 | 0.113 ± 0.000 |
| Slide Coefficient, η | 0.551 ± 0.012 | 0.519 ± 0.019 | 0.501 ± 0.003 | 0.593 ± 0.014 |
| Axial Expansion (%) | 1.06 ± 0.04 | 0.91 ± 0.04 | 1.05 ± 0.07 | 1.07 ± 0.03 |
| Radial Expansion (%) | 0.21 ± 0.01 | 0.22 ± 0.02 | 0.21 ± 0.01 | 0.26 ± 0.00 |
| Ejection Pressure (psi) | 185 ± 6 | 201 ± 9 | 184 ± 10 | 201 ± 5 |
| Stripping Pressure (psi) | 96 ± 3 | 101 ± 5 | 99 ± 4 | 62 ± 3 |
| Green Density (g/cc) | 4.463 ± 0.000 | 4.453 ± 0.003 | 4.504 ± 0.006 | 4.234 ± 0.003 |
| Axial Green Strength (psi) | 216 ± 28 | 158 ± 5 | 190 ± 28 | 90 ± 23 |
| Radial Green Strength (psi) | 582* | ND [‡] | 848* | ND [‡] |
| Diametral Green Strength (psi) | 25 ± 2 | ND [‡] | 24* | 19 ± 1 |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table D-II. Compaction properties for the Dispersant / Alternate binder compacts at 6kpsi max pressure, dried

| Sample ID | Baseline | Baseline-Carbosperse | Baseline-DisperBYK | Aquazol-PEG6000 |
|---|---------------|----------------------|--------------------|-----------------|
| Moisture Content (wt.% binder) [†] | ~0.0 | ~0.4 | ~1.4 | ~0.0 |
| Compact Cohesiveness, c | 0.23 ± 0.02 | 0.23 ± 0.07 | 0.23 ± 0.02 | 0.12 ± 0.03 |
| Compactibility Coefficient, α | 0.128 ± 0.001 | 0.125 ± 0.002 | 0.129 ± 0.001 | 0.113 ± 0.000 |
| Slide Coefficient, η | 0.513 ± 0.015 | 0.514 ± 0.015 | 0.487 ± 0.006 | 0.539 ± 0.012 |
| Axial Expansion (%) | 1.28 ± 0.03 | 1.20 ± 0.09 | 1.21 ± 0.06 | 1.34 ± 0.04 |
| Radial Expansion (%) | 0.24 ± 0.02 | 0.25 ± 0.01 | 0.23 ± 0.02 | 0.29 ± 0.01 |
| Ejection Pressure (psi) | 167 ± 3 | 179 ± 1 | 164 ± 3 | 226 ± 16 |
| Stripping Pressure (psi) | 79 ± 4 | 80 ± 5 | 82 ± 4 | 73 ± 5 |
| Green Density (g/cc) | 4.433 ± 0.018 | 4.390 ± 0.029 | 4.454 ± 0.019 | 4.203 ± 0.009 |
| Axial Green Strength (psi) | 178 ± 4 | 176 ± 57 | 183 ± 11 | 89 ± 18 |
| Radial Green Strength (psi) | 1231* | 360* | 595* | ND [‡] |
| Diametral Green Strength (psi) | 29 ± 1 | 22 ± 1 | 14 ± 1 | 14* |

[†]wt.% normalized to binder and plasticizer content

[‡]strength is below the measurement threshold

*Only one data point could be collected, others were below measurement threshold

Table D-III. Compaction properties of the Dispersant / Alternate binder compacts at 40kpsi max pressure, humidified overnight

| Sample ID | Baseline | Baseline-Carbosperse | Baseline-DisperBYK | Aquazol-PEG6000 |
|---|---------------|----------------------|--------------------|-----------------|
| Moisture Content (wt.% binder) [†] | ~9.7 | ~8.0 | ~9.0 | ~4.3 |
| Compact Cohesiveness, <i>c</i> | 0.52 ± 0.06 | 0.48 ± 0.02 | 0.41 ± 0.05 | 0.19 ± 0.13 |
| Compactibility Coefficient, <i>α</i> | 0.135 ± 0.000 | 0.134 ± 0.000 | 0.135 ± 0.000 | 0.126 ± 0.000 |
| Slide Coefficient, <i>η</i> | 0.643 ± 0.004 | 0.635 ± 0.003 | 0.638 ± 0.003 | 0.652 ± 0.004 |
| Axial Expansion (%) | 3.34 ± 0.03 | 3.29 ± 0.03 | 3.17 ± 0.06 | 3.33 ± 0.06 |
| Radial Expansion (%) | 0.61 ± 0.02 | 0.62 ± 0.02 | 0.60 ± 0.01 | 0.66 ± 0.01 |
| Ejection Pressure (psi) | 1687 ± 70 | 1735 ± 20 | 1346 ± 36 | 1763 ± 107 |
| Stripping Pressure (psi) | 807 ± 22 | 841 ± 25 | 720 ± 30 | 490 ± 29 |
| Green Density (g/cc) | 5.023 ± 0.003 | 5.012 ± 0.004 | 5.038 ± 0.005 | 4.868 ± 0.006 |
| Axial Green Strength (psi) | 1921 ± 200 | 1827 ± 86 | 1520 ± 195 | 903 ± 228 |
| Radial Green Strength (psi) | 1323 ± 159 | 1778 ± 435 | 1393 ± 250 | 2035 ± 201 |
| Diametral Green Strength (psi) | 45 ± 8 | 67 ± 24 | 88 ± 42 | 77 ± 28 |

[†]wt.% normalized to binder and plasticizer content

Table D- IV. Compaction properties of the Dispersant / Alternate binder compacts at 40kpsi max pressure, dried.

| Sample ID | Baseline | Baseline-Carbosperse | Baseline-DisperBYK | Aquazol-PEG6000 |
|---|---------------|----------------------|--------------------|-----------------|
| Moisture Content (wt.% binder) [†] | ~0.0 | ~0.4 | ~1.4 | ~0.0 |
| Compact Cohesiveness, c | 0.72 ± 0.06 | 0.58 ± 0.05 | 0.70 ± 0.05 | 0.28 ± 0.01 |
| Compactibility Coefficient, α | 0.131 ± 0.001 | 0.131 ± 0.001 | 0.132 ± 0.001 | 0.124 ± 0.001 |
| Slide Coefficient, η | 0.687 ± 0.007 | 0.677 ± 0.009 | 0.666 ± 0.005 | 0.643 ± 0.002 |
| Axial Expansion (%) | 3.31 ± 0.09 | 3.48 ± 0.07 | 3.61 ± 0.03 | 3.55 ± 0.28 |
| Radial Expansion (%) | 0.63 ± 0.02 | 0.63 ± 0.02 | 0.63 ± 0.02 | 0.69 ± 0.01 |
| Ejection Pressure (psi) | 1356 ± 10 | 1400 ± 8 | 1183 ± 15 | 2187 ± 114 |
| Stripping Pressure (psi) | 500 ± 17 | 521 ± 30 | 476 ± 32 | 604 ± 31 |
| Green Density (g/cc) | 4.972 ± 0.014 | 4.954 ± 0.015 | 4.960 ± 0.011 | 4.823 ± 0.024 |
| Axial Green Strength (psi) | 2315 ± 200 | 1927 ± 134 | 2420 ± 147 | 1033 ± 57 |
| Radial Green Strength (psi) | 1633 ± 414 | 1769 ± 255 | 1404 ± 456 | 2585 ± 559 |
| Diametral Green Strength (psi) | 88 ± 20 | 77 ± 18 | 82 ± 19 | 52 ± 4 |

[†]wt.% normalized to binder and plasticizer content

APPENDIX E. DISPERSANT / ALTERNATE BINDER COMPACTION PROPERTY GRAPHS

The Baseline in all graphs is the baseline powder, humidified, at either 6kpsi or 40kpsi.

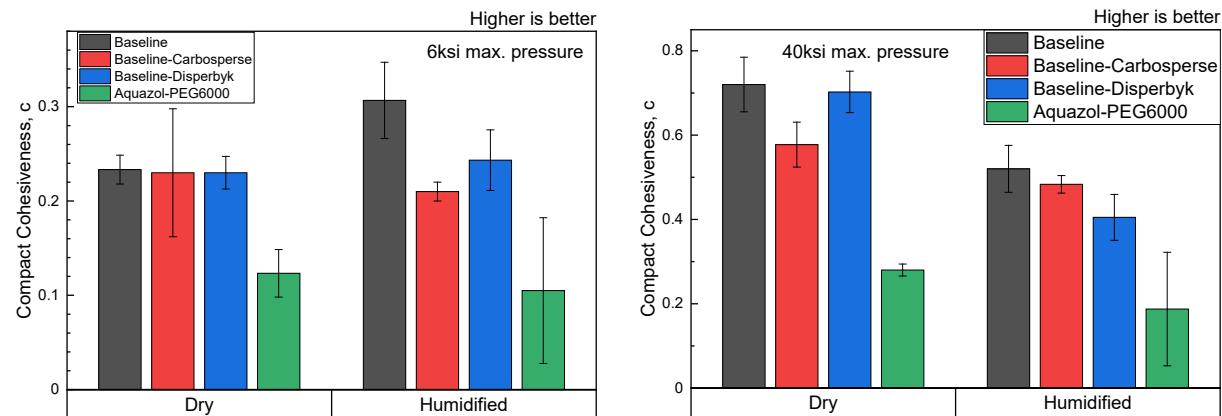


Figure E-1. Compact cohesiveness for the Dispersant / Alternate binder compacts.

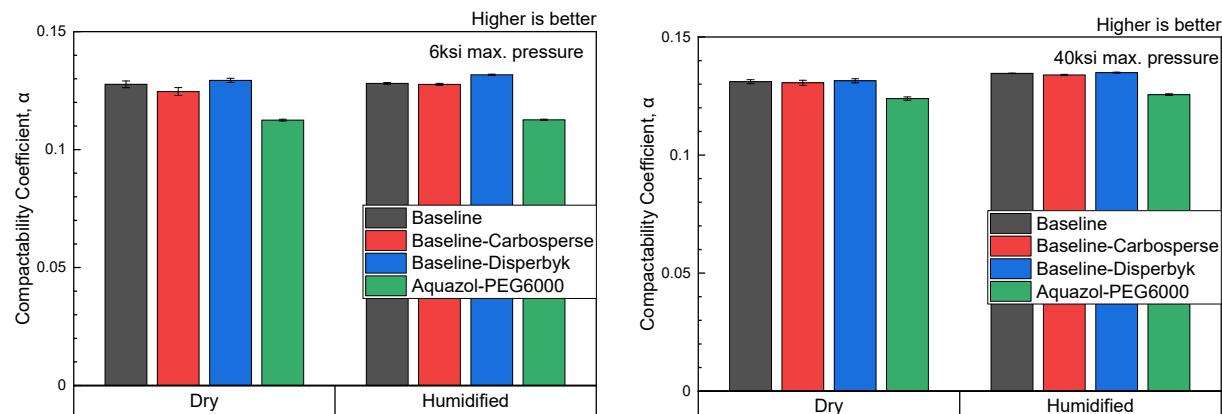


Figure E-2. The compactability for the Dispersant / Alternate binder compacts.

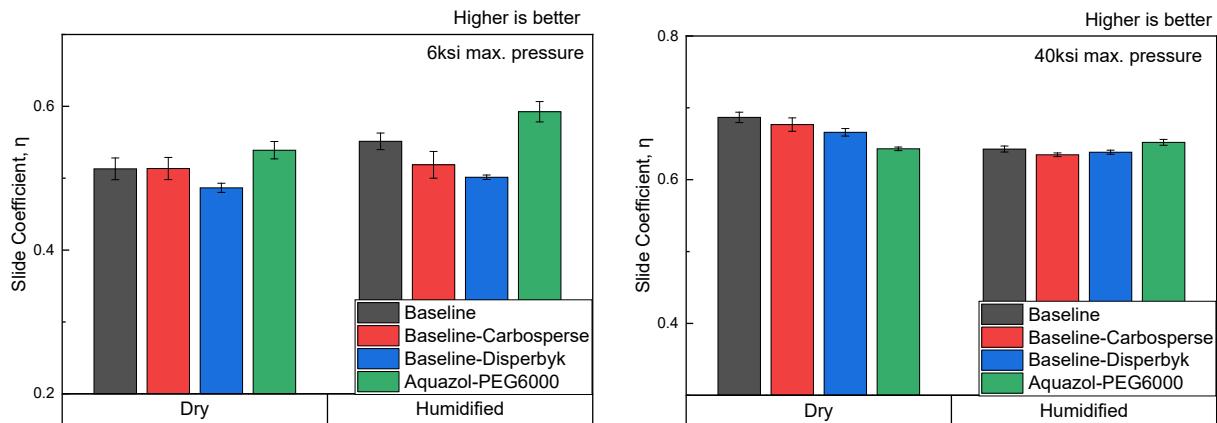


Figure E-3. Slide coefficient of the Dispersant / Alternate binder compacts.

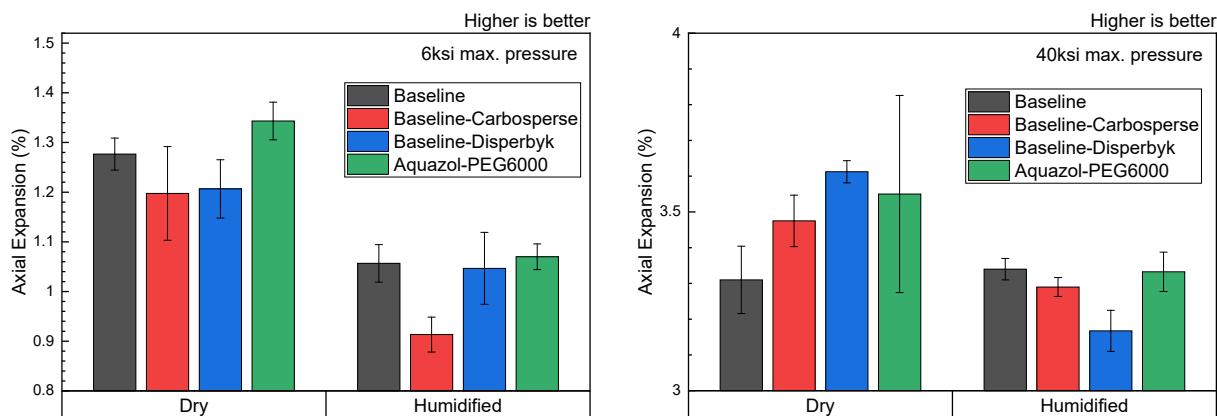


Figure E-4. Axial expansion of the Dispersant / Alternate binder compacts.

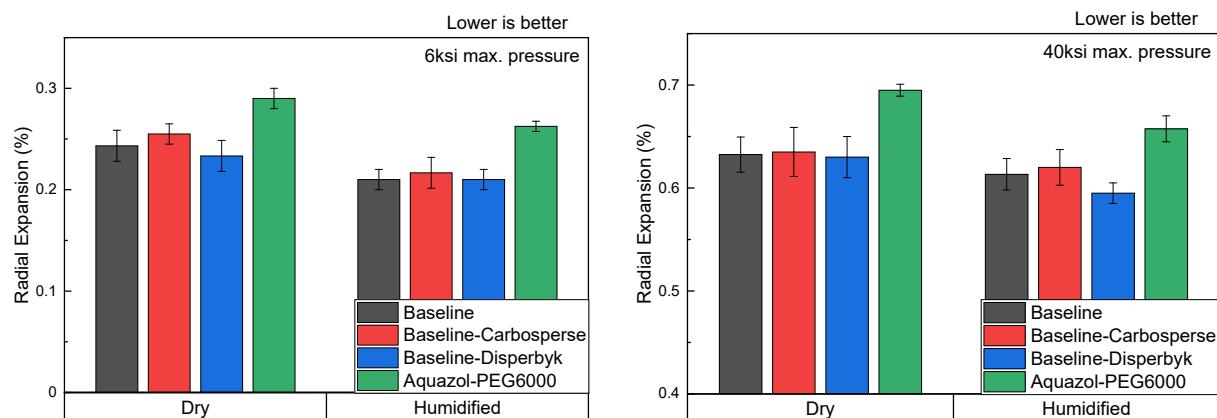


Figure E-5. Radial expansion of the for the Dispersant / Alternate binder compacts.

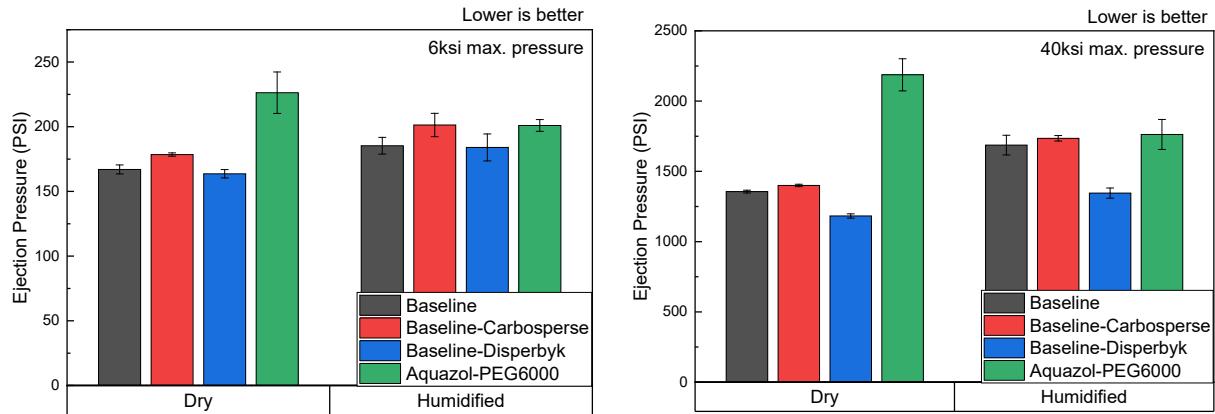


Figure E-6. Ejection pressure for the Dispersant / Alternate binder compacts.

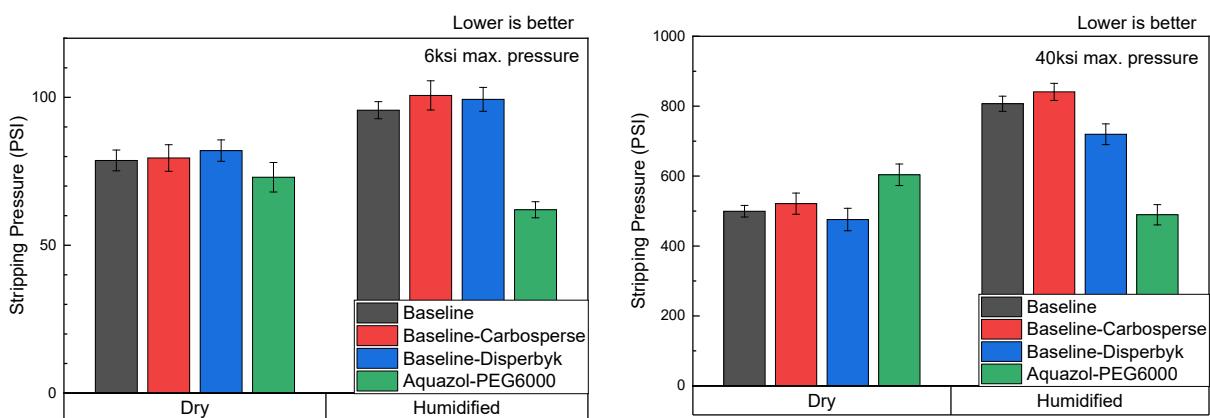


Figure E-7. Stripping pressure for the Dispersant / Alternate binder compacts.

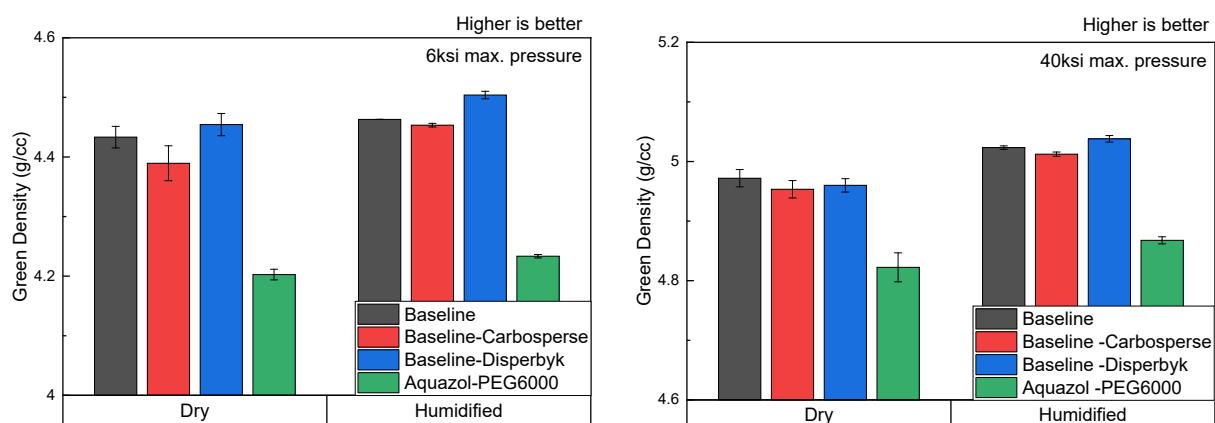


Figure E-8. Out-of-die green density for the Dispersant / Alternate binder compacts.

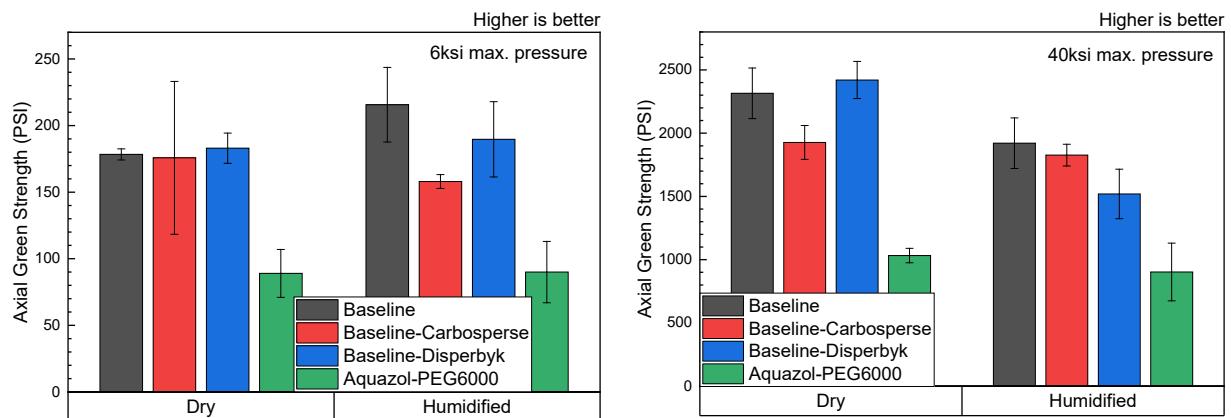


Figure E-9. Axial green strength for the Dispersant / Alternate binder compacts.

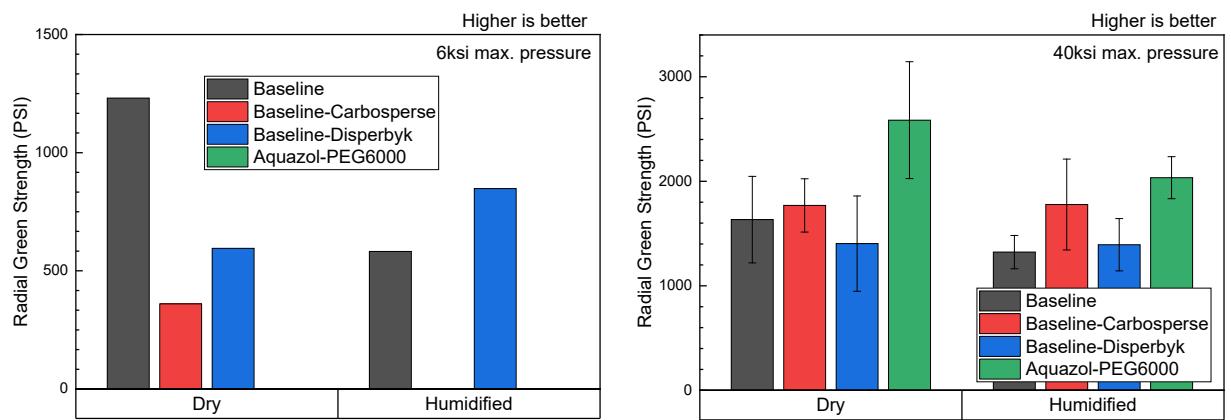


Figure E-10. Radial green strength for the Dispersant / Alternate binder compacts.

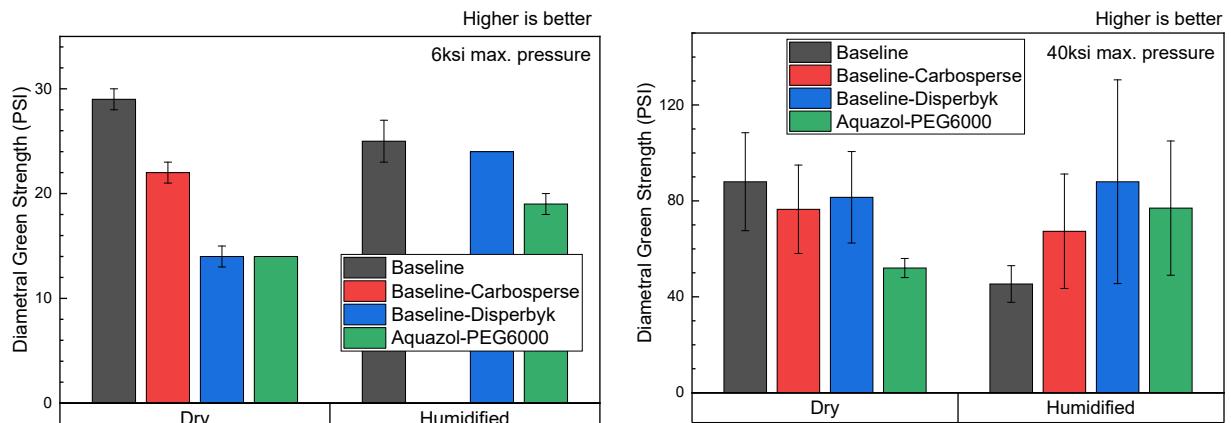


Figure E-11. Diametral green strength for the Dispersant / Alternate binder compacts.

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