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Comparison of the elastic recovery and strain-in-compression of commercial and novel vinylpoly siloxane impression materials incorporating a novel crosslinking agent and a surfactant

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The aim of the study was to formulate the experimental vinyl polysiloxane (VPS) impression materials and to compare its elastic recovery and strain-in-compressions with three commercial VPS materials (Aquasil, Elite, and Extrude). Experimental (Exp) materials, two hydrophobic (Exp-I and II) and three hydrophilic (Exp-III, IV and V) were developed. Exp 1 was based on vinyl-terminated poly-dimethylsiloxane and a conventional cross-linking agent (poly methylhydrosiloxane), while Exp II contained a novel cross-linking agent tetra-functional (dimethylsilyl) ortho-silicate (TFDMSOS). Hydrophilic materials were formulated by incorporating different concentrations of non-ionic surfactant (Rhodasurf CET-2) into Exp II formulation. Measurement of elastic recovery and strain-in-compression for commercial and experimental VPS were performed according to ISO4823 using the calibrated Tenius Olsen. One way Analysis of Variance (one way ANOVA) and Tukey's post-hoc (HSD) test were used for statistical analysis and a p-value of ≤ 0.05 was considered significant. It was noted that Exp-II (containing TFDMSOS) exhibited the highest elastic recovery. Exp-I has statistically similar values to commercial VPS. Addition of the Rhodasurf CET-2 reduces the % elastic recovery and the % reduction was directly related to the concentration of Rhodasurf CET-2. However, all experimental materials had greater values than commercial materials. It was also noted Exp II (TFDMSOS containing) had

significantly higher strain-in-compression values compared to Exp-I and commercial materials. These values displayed a further significant increase when a non-ionic surfactant (Rhodasurf CET-2) was added (Exp-III, IV and V).

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Abstract

The aim of the study was to formulate the experimental vinyl polysiloxane (VPS) impression materials and to compare its elastic recovery and strain-in-compressions with three commercial VPS materials (Aquasil, Elite, and Extrude). Experimental (Exp) materials, two hydrophobic (Exp-I and II) and three hydrophilic (Exp-III, IV and V) were developed. Exp 1 was based on vinyl-terminated poly-dimethylsiloxane and a conventional cross-linking agent (poly methylhydrosiloxane), while Exp II contained a novel cross-linking agent tetra-functional (dimethylsilyl) ortho-silicate (TFDMSOS). Hydrophilic materials were formulated by incorporating different concentrations of non-ionic surfactant (Rhodasurf CET-2) into Exp II formulation. Measurement of elastic recovery and strain-in-compression for commercial and experimental VPS were performed according to ISO4823 using the calibrated Tenius Olsen. One way Analysis of Variance (one way ANOVA) and Tukey's post-hoc (HSD) test were used for statistical analysis and a p-value of ≤ 0.05 was considered significant. It was noted that Exp-II (containing TFDMSOS) exhibited the highest elastic recovery. Exp-I has statistically similar values to commercial VPS. Addition of the Rhodasurf CET-2 reduces the % elastic recovery and the % reduction was directly related to the concentration of Rhodasurf CET-2. However, all experimental materials had greater values than commercial materials. It was also noted Exp II

(TFDMSOS containing) had significantly higher strain-in-compression values compared to Exp-I and commercial materials. These values displayed a further significant increase when a non-ionic surfactant (Rhodasurf CET-2) was added (Exp-III, IV and V).

Introduction

Dental impression refers to a negative imprint of oral hard and soft tissues. It is used to fabricate a positive replica (cast) on which the indirect prosthesis can be fabricated. Therefore, an accurate impression is of utmost importance for the successful fabrication of a prosthesis. Ideally, the impression material should have good wettability, accuracy, elasticity and minimal distortion on removal and storage. Impression materials are compressed against the tray, especially while recording undercut areas, and on the removal of impression from the mouth. The degree of distortion of the material depends on the severity of the undercut, elastic recovery of the material, the time the material is kept in the compressed state and storage conditions

The elastic recovery of impression material is the capacity of the material to revert to its original position, without significant distortion after being strained, when the deforming force is removed [1]. It is due to the presence of folded polymeric segments within the material, which coil and uncoil during loading and unloading. Therefore, the greater the elastic recovery of the material, the more precise the prosthesis. It has been reported that the likelihood of permanent deformation increases on slow removal of an impression as the material is stressed for longer duration [2-7]. None of the impression materials has 100% elastic recovery[1], rather most elastomeric materials exhibit time-dependent recovery from deformation (viscoelasticity) [8-10].

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74 The elastic recovery of these materials depends on their composition, such as the pre-polymer,
75 cross-linking agents, and fillers [5,11-16]. International Standards Organisation (ISO) 4823 [17],
76 recommends that an elastomeric impression material (all consistencies) must have 96.5% elastic
77 recovery. Although all elastomeric impression materials fulfil the criteria, the VPS possesses better
78 elastic recovery compared to other impression materials [1,5,13,18]. This allows pouring of the
79 impression to fabricate cast after six minutes of removal from the mouth [19]. In addition, these
80 materials exhibit great dimensional stability and high tear strength.

81

82 Different brands of VPS impression materials showed variations in elastic recovery. Lawson,
83 Burgess, Litaker [11] investigated the elastic recovery for five VPS and a hybrid impression material
84 (containing siloxane and polyether groups) after subjecting materials to tensile and compressive
85 stress. The VPS impression materials showed improved elastic recovery in comparison to the
86 hybrid material, which may be related to the compositions of materials as hybrid material
87 composed of polyethers, which have a lower elastic recovery compared to VPS [3,20].

88

89 Strain-in-compression is the measurement of the stiffness or flexibility of impression material. It
90 determines the ability of polymerized material to be removed from the mouth or cast without
91 permanent deformation, injury to oral tissues or fracture. Also, it dictates the ability of the
92 impression to resist deformation and withstand the weight of the dental stone when the cast is
93 poured [13,21,22].

To overcome the problem of inherent hydrophobicity of VPS and to improve tear strength and % elongation at break of the material, in our previous work, novel formulations of VPS were fabricated using a novel cross-linking agent i.e. tetra-functional (dimethylsilyl) ortho-silicate (TFDMSOS) and novel surfactant i.e. Rhodasurf CET-2 [5,15,23]. Different researchers have explored the effect of various surfactants to improve the hydrophilicity of the material, however, little work has been carried out to improve the tear strength of VPS impressions. Additionally, the effects of the addition of surfactant on the mechanical properties of the materials and the hydrophilicity of these modified materials after disinfection requires further exploration.

Ud Din et al. observed that the incorporation of a novel cross-linking agent (TFDMSOS) significantly improved the materials' % elongation-at-break and tear strength compared to the control containing a conventional crosslinking agent-poly (methyl-hydro siloxane) [5]. Additionally, the incorporation of a novel surfactant (Rhodasurf CET-2) further resulted in a significant increase in % elongation-at-break [15]. It was also noted that the experimental formulation has a lower contact angle (improved hydrophilicity) than commercial formulations. Additionally, disinfection has little impact on the contact angle as the surfactant did not readily leach out in a disinfecting solution [23]. However, mechanical testing including elastic recovery and strain-in-compression required further exploration before considering the experimental formulation as a better alternative to commercially available VPS impression materials.

The purpose of this study was to evaluate the effect of a novel cross-linking agent, TFDMSOS and novel surfactant (Rhodasurf CET-2) on the elastic recovery and strain-in-compression of

experimental VPS and to compare it with commercial materials. It was hypothesized that experimental formulations have better elastic recovery and strain-in compression-values, making it a more suitable material for recording an accurate impression.

Materials & Methods

Three medium-body commercial VPS impression materials were used in this study; Aquasil Ultra Monophase, USA, Dentsply (Aq M), Elite HD Monophase, Italy, Zhermack (Elt M) and Extrude, USA, Kerr (Extr M). Additionally, five experimental VPS formulations were prepared as base paste and catalyst paste (Table 1). Exp-I was used as a control for Exp-II, while Exp-II acted as a control group for Exp-III, Exp-IV and Exp-V.

Preparation of Experimental Formulations

The base paste of Exp-1 (hydrophobic VPS) was formulated by mixing vinyl-terminated poly-dimethylsiloxane and a conventional cross-linking agent (poly methylhydrosiloxane) for 5 minutes using an electric hand mixer (Kenwood, kMix, UK). The filler (Aerosil R812S) was added to the mixture and a uniform paste was made by mixing the components with a pestle and mortar for 5 min, followed by blending the paste with an electric mixer for 10 minutes. The catalyst paste was formulated by mixing vinyl-terminated poly (dimethylsiloxane), platinum catalyst and palladium for 5 minutes with the electric hand mixer, followed by the addition of filler (Aerosil R812S) and mixing it with the pestle and mortar and electric hand mixer.

Exp-II was prepared by reducing the amount of conventional crosslinking agent poly (methylhydrosiloxane), from 1.10% to 0.77% and replacing it with a novel cross-linking agent

(TFDMSOS) in the base paste of Exp-I formulation while maintaining 1:1 ratio of vinyl to silane groups. The catalyst paste for Exp-II was similar to that of Exp-I.

The base-paste of Exp-II was further modified by the addition of a novel surfactant (Rhodasurf CET-2) in concentrations of 2%, 2.5% and 3% to form experimental formulations III, IV and V respectively. Due to the addition of a surfactant in the base paste, the quantities of the constituents in the catalyst paste were modified to ensure adequate polymerization of the materials (Table 1). The prepared base and catalyst paste of experimental materials were kept in separate compartments of cartridge and stored at 4ºC ± 2ºC.

Sample preparation for elastic recovery and strain under compression

The cylindrical samples for elastic recovery and strain-in-compression were prepared using polytetrafluoroethylene (PTFE) mould measuring 20 mm in length x 12.5 mm in diameter according to ISO4823 [17] standard. PTFE mould was positioned on top of a metal plate lined with an acetate sheet. The base and catalyst pastes were syringed into the mould using an auto-mixing syringe and the mould was sandwiched by another acetate-lined metal plate. It helped in removing excess material, and to ensured formation of flat smooth surface of the specimen. The assembly was held using C-clamp. Commercial materials were left to polymerize according to the manufacturer's instructions while experimental materials were allowed to be set for 4 to 11 minutes[23].

Twelve samples per formulation were prepared for estimating elastic recovery. Two metal plates (13 x 13 x 3 mm^3) were fixed on either side of the specimen with the aid of double-sided sticky tape. The length of the specimen (including metal plates) was recorded (h_1) using a digital micrometre (Mitutoyo, Japan) to an accuracy of 0.001mm. Then the specimen was deformed to 6±0.1 mm within 1 second using the calibrated Tinius Olser. (Figure 1). The deformation force

was released slowly over a period of 5 seconds. After two minutes the length was measured again (h_2). The elastic recovery in percentage, K, was assessed using equation 1.

$$\textbf{Equation 1: } \mathbf{K = 100 - \left[100\left(\frac{h_1 - h_2}{ho}\right)\right]}$$

h_0 is the height (mm) of the split mould

h_1 is the length (mm) of the specimen immediately before the application of the initial load

h_2 is the length of the specimen, 2 minutes after removing the deformation force

To evaluate strain-in-compression, 12 samples per material were tested. An initial force of 1.22±0.1 N was exerted on the specimen and the distance (h_1) was calculated using the Tinius Olsen (Figure 1). The load was increased to 12.25±0.1 (N) progressively over a time of 10 seconds at a rate of 3 mm/min and a change in height of the specimen was noted again (h_2). The percentage of strain-in-compression, E, was calculated using equation 2.

$$\textbf{Equation 2: } \mathbf{E = \left(\frac{h_1 - h_2}{h_0}\right)100}$$

h_0 is the height (mm) of the split mould

h_1 is the length (mm) of the specimen, 30 seconds after submission of the opening load

h_2 is the length of the specimen, 30 seconds after submission of the amplified load.

The data was analyzed using SPSS Version 22 (Armonk NY IBM Corp, Armonk, NY, USA). Numerical data were presented as mean and standard deviation. Analysis of variance was

183 performed with p value at 0.05. Where significant difference in group was found, individual
184 means were compared using post hoc Tukey's test.
185

186 ## Results

187 Table 2 shows the elastic recovery and strain-in-compression for commercial and experimental
188 VPS impression materials immediately after setting. All the tested materials met the ISO4823
189 requirement of having elastic recovery greater than 96.5%. Exp-II exhibited the highest elastic
190 recovery while Exp-V demonstrated the lowest values. The post-hoc analysis revealed that all
191 three commercial products and Exp-I had statistically similar elastic recoveries.
192

193 Utilization of novel crosslinking agent (TFDMSOS) instead of conventional agent significantly
194 increased elastic recovery. A significant difference in the elastic recovery was noted between Exp-
195 II and Exp-V. It was noted that the addition of a non-ionic surfactant (Rhodasurf CET-2) in the
196 experimental formulation, to improve hydrophilicity of material, resulted in a reduced percentage
197 of elastic recovery of material, however, the changes were statistically not significant (Table 2).
198

199 **Strain-in-compression**

200 Figure 2 and Table 2 reveal the strain-in-compression for the tested VPS impression materials.
201 Experimental VPS had significantly higher (at $p < 0.05$) strain-in-compressions values compared
202 to the commercial VPS. Exp-V exhibited significantly the highest (Tukey's HSD test) strain-in-
203 compression (7.08 % ± 0.22 %) while Elt M had the lowest values (3.15 % ± 0.18 %). Among
204 commercial materials, no significant difference between Aq M and Extr M was noted. However,
205 it was noted that the addition of a novel crosslinking agent i.e., TFDMSOS (Exp II), significantly

increased the percentage strain-in-compression values compared to formulations based on conventional cross-linking agents (Exp-I, Aq M, Elt M, Extr M). Also, it was observed that experimental formulations incorporating non-ionic surfactant (Rhodasurf CET-2) led to a further significant increase in strain-in-compression values and this effect was concentration dependant.

Figure 3 and Table 2 show the comparison between elastic recovery and strain-in-compression for all commercial and Experimental VPS impression materials evaluated in this study. Among the experimental materials, there is a correlation between elastic recovery and strain-in-compression. With the addition of TFDMSOS in Exp-II the elastic recovery and strain-in-compression increase significantly compared to Exp-I (control). However, there is a negative correlation seen after addition of Rhodasurf CET-2 (non-ionic surfactant) in Exp III. With the addition of surfactant the elastic recovery is deceased while stain-in-compression is increased. It can also be noticed that with the increase in % amount of surfactant there is a consistent and significant decrease in elastic recovery and significant increase in strain-in-compression in Exp-IV and Exp-V. Among commercial materials, no significant difference were seen.

Discussion

The elastic recovery of the impression materials play a major role in the accurate reproduction of the oral cavity. The ability of elastomeric impression materials to revert to their actual form upon removal of the applied stress is related to their coiled wrapped and kinked molecular chains. These polymer chains stretch in the direction of stress and, recoil back on releasing the force, gaining their original shape and form [1,13]. In the present study, elastic recovery and strain-in-compression of commercial and experimental VPS impression materials were compared.

Values for elastic recovery for commercial and experimental VPS impression materials ranged from 99.11 to 99.64%. These values were within the range set by International Standards Organization (ISO) 4823 (2007) which requires ≥96.5 %. Similar results were reported by Lawson, Burgess, Litaker [11], who noted that elastic recovery of five tested VPS (Aquasil Ultra, Examix, Genie, Imprint 3, and StandOut) and one hybrid impression material (Senn) ranges from 99.34 to 99.83 %. In another study Lu, Nguyen, Powers [22] investigated the elastic recovery of two VPS (Flexitime and Imprint II) and one polyether (Impregum). It was noted that Flexitime, Imprint II and Impregum had 99.60, 99.75 and 99.19 % elastic recoveries respectively.

~~Statistically~~ similar percentage elastic recovery was noted for the commercial materials and Exp-I containing conventional cross-linking agent (polymethylhydrosiloxane). However, on incorporating a novel cross-linking agent (TFDMSOS) in Exp-II, there was a ~~statistical~~ increase in % elastic recovery. The greater elastic recovery of Exp-II is attributed to excellent crosslinking of TFDMSOS with functional groups of vinyl-terminated poly (dimethylsiloxane) pre-polymer as each molecule of TFDMSOS can bond to four functional groups of pre-polymer [15]. Similar results have been reported in the literature indicating the amount of permeant deformation of an impression material is greatly influenced by the degree of cross-linking of the polymeric chains [24]. The degree of polymerization also affects other mechanical properties of elastomeric impression materials such as tear strength and % elongation-at-break [5,15]. reported improved tear strength and % elongation-at-break due to increased cross-linking of polymeric chains

The strain-in-compression was also calculated to assess the rigidity of impression materials so that it can be removed from the mouth or cast without permanent deformation after setting, and to resist deformation when the dental stone is poured. All tested impression materials have values for ~~%~~ strain-in-compression within the ISO4823 [17] limits. Experimental VPS impression materials had

higher strain-in-compression values indicating improved flexibility of experimental material [25]. Therefore, a positive correlation between the degree of polymerization, elastic recovery and strain-in-compression was noted (Figure 3 and Table 2). Additionally, it was observed that the incorporation of the wetting agent (Rhodasurf CET-2), further significantly increased strain-in-compression values. This was contradictory to the results of Lu et al. who noticed that flexible materials have less cross-linking and have better elastic recovery. This might be due to the difference in the composition of the materials used in the present study.

The ability to undergo greater elastic recovery is a desirable property of impression materials as it ensures an accurate impression which in turn guarantees a correct fit of the prosthesis. The experimental VPS impression materials in this study show greater elastic recovery than their commercial counterparts. Additionally, in previous studies, same experimental material has proven to have improved wettability, percentage elongation, tear strength and minimal distortion after disinfection making them a much more suitable option for impression taking [5,6,15].

A limitation of this study is that it is conducted in an in-vitro environment under laboratory conditions. To strengthen the claim of experimental VPS as a superior impression material, it is necessary to conduct further research in intra-oral, in-vivo, conditions.

Conclusions

Within the limitations of this study, the addition of a novel cross-linking agent (TFDMSOS) showed improved elastic recovery and strain-in-compression, while the addition of a non-ionic surfactant also significantly increased strain-in-compressions values for all experimental VPS. All tested materials comply with ISO standards. In the future, it is recommended that clinical

investigations should be undertaken, and material selection should be based on adequate knowledge of the properties of materials to improve clinical success.

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Table 1(on next page)

Composition of novel experimental (Exp-I, II, III, IV and V) VPS impression materials.

1 **Table 1**. Composition of novel experimental (Exp-I, II, III, IV and V) VPS impression materials.

2

Components	Base Paste (Wt %)				
	Exp-I	Exp-II	Exp-III	Exp-IV	Exp-V
Vinyl-terminated dimethylpolysiloxane (Mw 62700)	39.90	39.90	37.95	37.46	36.98
Polymethylhydrosiloxane (Mw 2270)	1.10	0.77	0.74	0.73	0.72
Tetra-functional (dimethylsilyl) orthosilicate (TFDMSOS) (Mw 329)	-	0.33	0.32	0.31	0.31
Filler Aerosil R 812	9	9	9	9	9
Components	Catalyst Paste (Wt %)				
	Exp-I	Exp-II	Exp-III	Exp-IV	Exp-V
Vinyl-terminated dimethylpolysiloxane (Mw 62700)	40.72	40.72	39.51	39.51	39.51
Platinum (0.05 M)	0.06	0.06	1.27	1.27	1.27
Palladium (<1μm)	0.23	0.23	0.22	0.22	0.22
Filler Aerosil R 812	9	9	9	9	9
Rhodasurf CET-2 (non-ionic surfactant)	-	-	2.00	2.50	3.00

Table 2(on next page)

Average elastic recovery and strain in compression of commercial and experimental VPS immediately after setting.

Table 2. Average elastic recovery and strain in compression of commercial and experimental VPS immediately after setting.

Impression Materials	Elastic Recovery (%)	Strain-in-compression
Aq M	99.32 ± 0.30	4.261±0.154
Elt M	99.31 ± 0.35	3.153±0.177
Extr M	99.27 ± 0.32	4.405±0.118
Exp-I	99.32 ± 0.52	4.677±0.207
Exp-II	99.65 ± 0.09	5.360±0.163
Exp-III	99.50 ± 0.23	6.137±0.256
Exp-IV	99.37 ± 0.26	6.541±0.239
Exp-V	99.12 ± 0.16	7.076±0.220

Figure 1

Tinnus Oslen assembly used to determine elastic recovery and strain-in-compression of experimental and commercial polymeric vinylpoly siloxane impression materials.

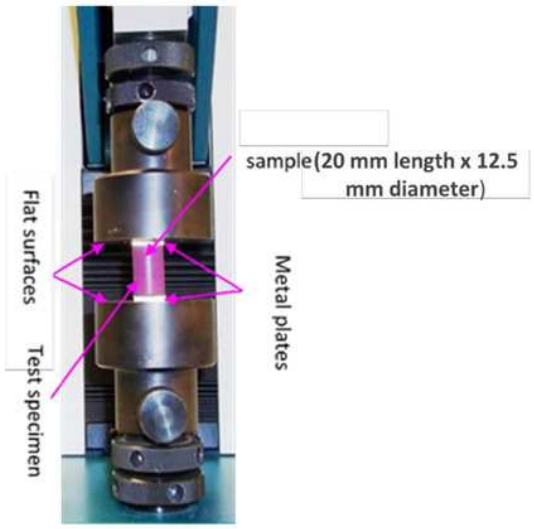

Figure 1: Tinnus Oslen assembly used to determine elastic recovery and strain-in-compression of experimental and commercial polymeric vinylpoly siloxane impression materials.

Figure 2

Mean (± standard errors; n=12) strain-in-compression of commercial and experimental polymeric VPS immediately after setting. Similar letters indicate no significant difference (p˃0.05).

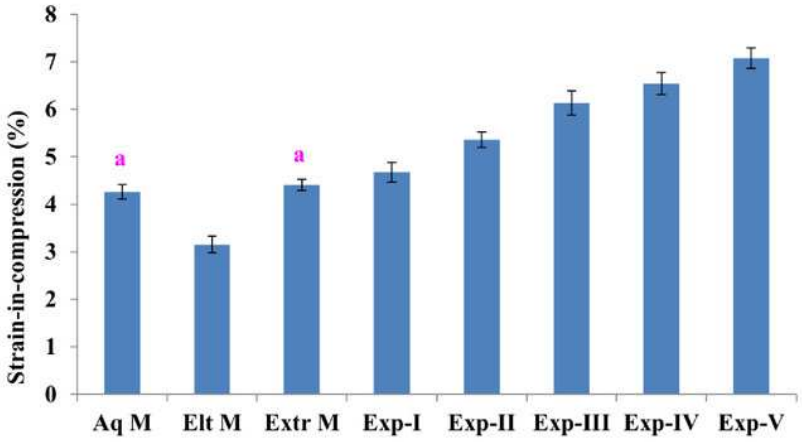

Figure 2. Mean (± standard errors; n=12) strain-in-compression of commercial and experimental polymeric VPS immediately after setting. Similar letters indicate no significant difference ($p>0.05$).

Figure 3

Mean (± standard errors; n=12) comparison of elastic recovery and strain-in-compression of commercial and experimental VPS immediately after setting.

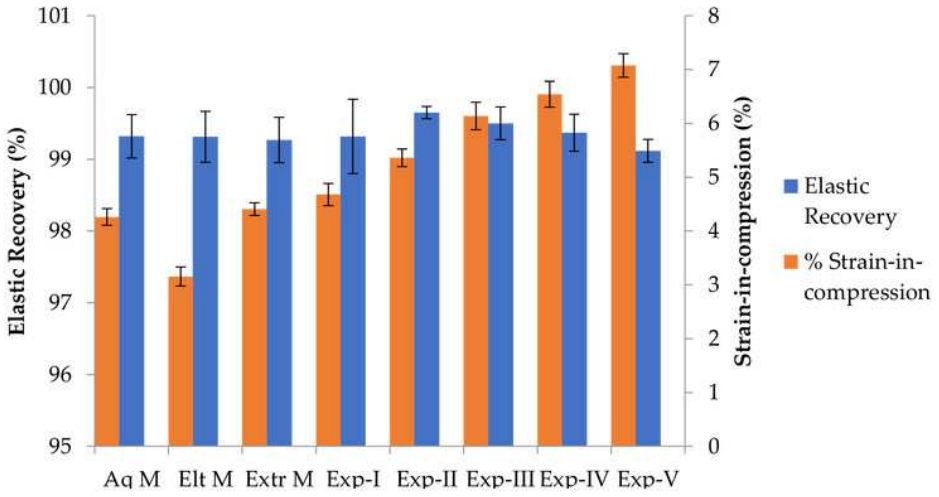

Figure 3. Mean (± standard errors; n=12) comparison of elastic recovery and strain-in-compression of commercial and experimental VPS immediately after setting.