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

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Abstract

This study aims to formulate experimental vinyl polysiloxane (VPS) impression materials and compare their elastic recovery and strain-in-compressions with three commercial VPS materials (Aquasil, Elite, and Extrude). Five experimental materials (Exp), two hydrophobic (Exp-I and II) and three hydrophilic (Exp-III, IV and V) were developed. Exp 1 contained vinyl-terminated poly-dimethyl siloxane and a conventional cross-linking agent (poly methylhydrosiloxane), while Exp-II contained a novel cross-linking agent that is tetra-functional dimethyl-silyl-ortho-silicate (TFDMSOS). Exp III-V (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 materials were performed according to ISO4823 standard using the calibrated mechanical testing machine (Tinius Olsen). One-way Analysis of Variance (one-way ANOVA) and Tukey's posthoc (HSD) test were used for statistical analysis and a p-value of ≤ 0.05 was considered significant. Exp-I has statistically similar values to commercial VPS. The Exp-II showed the highest elastic recovery, while % elastic recovery was reduced with the addition of the non-ionic surfactant (Rhodasurf CET-2). The % reduction was directly related to the concentration of Rhodasurf CET-2. In addition, Exp II had significantly higher strain-in-compression values compared to Exp-I and commercial materials. These values were further increased with the addition of a non-ionic surfactant (Rhodasurf CET-2) was added (Exp-III, IV and V).

Introduction

54 Dental impression refers to a negative imprint of oral hard and soft tissues [1]. An accurate

55 impression is of utmost importance for the successful fabrication of a prosthesis. Ideally, the

56 impression material should have good wettability, accuracy, elasticity and minimal distortion on

57 removal and storage [1]. Impression materials are compressed against the tray, especially while

58 recording undercut areas, and on the removal of impression from the mouth. The degree of

59 distortion of the material depends on the severity of the undercut, elastic recovery of the material,

60 the time the material is kept in the compressed state and storage conditions [1,2].

61

62 The elastic recovery of impression material is the capacity of the material to revert to its original

63 position, without significant distortion after being strained, when the deforming force is removed

64 (Hamalian et al. 2011). It is due to the presence of folded polymeric segments within the material,

65 which coil and uncoil during loading and unloading. Therefore, the greater the elastic recovery of

66 the material, the more precise the prosthesis.

67

68 The likelihood of permanent deformation increases on slow removal of an impression as the

69 material is stressed for longer duration (Balkenhol et al. 2010; Din et al. 2018b; Din et al. 2021;

70 Din et al. 2022; Hondrum 1994; Mandikos 1998). None of the impression materials has 100%

71 elastic recovery(Hamalian et al. 2011), rather most elastomeric materials exhibit time-dependent

72 recovery from deformation (viscoelasticity) (Braden et al. 1997; Darvell 2009; Goldberg 1974).

73 The elastic recovery of these materials depends on their composition, such as the pre-polymer,

74 cross-linking agents, and fillers (Deb 1998; Din et al. 2018a; Din et al. 2018b; Klooster et al. 1991;

75 Lawson et al. 2008; Saeed et al. 2022; Ud Din et al. 2018).

76

77 International Standards Organisation (ISO) 4823 (2007), recommends that an elastomeric

78 impression material (all consistencies) must have 96.5% elastic recovery. Although all elastomeric

79 impression materials fulfil the criteria, the VPS possesses better elastic recovery compared to other

80 impression materials (Bonsor & Pearson 2013; Din et al. 2018b; Hamalian et al. 2011; Klooster et

81 al. 1991). This allows pouring of the impression to fabricate cast after six minutes of removal from

82 the mouth (Blomberg et al. 1992). In addition, these materials exhibit great dimensional stability

83 and high tear strength.

84

85 Different brands of VPS impression materials showed variations in elastic recovery. Lawson et al.

86 (2008) investigated the elastic recovery for five VPS and a hybrid impression material (containing

87 siloxane and polyether groups) after subjecting materials to tensile and compressive stress. The

88 VPS impression materials showed improved elastic recovery in comparison to the hybrid material,

89 which may be related to the compositions of materials as hybrid material composed of polyethers,

90 which have a lower elastic recovery compared to VPS (Hondrum 1994; Ud Din et al. 2022a; Ud

91 Din et al. 2022b).

92

93 Strain-in-compression is the measurement of the stiffness or flexibility of impression material. It

94 determines the ability of polymerized material to be removed from the mouth or cast without

95 permanent deformation, injury to oral tissues or fracture. Also, it dictates the ability of the

96 impression to resist deformation and withstand the weight of the dental stone when the cast is

97 poured (Helvey 2011; Klooster et al. 1991; Lu et al. 2004a).

98

99   To overcome the problem of inherent hydrophobicity of VPS and to improve tear strength and %

100 elongation at break of the material, in our previous work, novel formulations of VPS were

101 fabricated using a novel cross-linking agent i.e. tetra-functional (dimethylsilyl) ortho-silicate

102 (TFDMSOS) and novel surfactant i.e. Rhodasurf CET-2 (ethoxylated cetyloleyl alcohol (Din et al.

103 2018a; Din et al. 2018b; Din et al. 2017). The addition of TFDMOS improved mechanical

104 properties of experimental impression materials, while the non-ionic surfactant was added to

105 improve wetting properties of the materials. Different researchers have explored the effect of

106 various surfactants to improve the hydrophilicity of the material, however, little work has been

107 carried out to improve the tear strength of VPS impressions. Additionally, the effects of the

108 addition of surfactant on the mechanical properties of the materials and the hydrophilicity of these

109 modified materials after disinfection requires further exploration. Rhodasurf CET-2 is a non-ionic

110 surfactant which is made by combination of ethoxylated cetyl and ethoxylated oleyl alcohols.

111 Ethoxylated oleyl alcohol has a double bond in its chemical structure. The double bond could be

112 possibly activated during mixing and played a vital role in the cross-linking polymerization

113 reaction leading to improved elastic recovery and strain-in-compression [23].

114 Ud Din et al. observed that the incorporation of a novel cross-linking agent (TFDMSOS)

115 significantly improved the materials' % elongation-at-break and tear strength compared to the

116 control containing a conventional crosslinking agent-poly (methyl-hydro siloxane) [5]. Additionally,

117 the incorporation of a novel surfactant (Rhodasurf CET-2) further resulted in a significant increase

118 in % elongation-at-break (Din et al. 2018a). It was also noted that the experimental formulation

119 has a lower contact angle (improved hydrophilicity) than commercial formulations. Additionally,

120 disinfection has little impact on the contact angle as the surfactant did not readily leach out in a

121 disinfecting solution (Din et al. 2017). However, mechanical testing including elastic recovery and

122   strain-in-compression required further exploration before considering the experimental

123   formulation as a better alternative to commercially available VPS impression materials.

124

125   The purpose of this study was to evaluate the effect of a novel cross-linking agent, TFDMSOS and

126   novel surfactant (Rhodasurf CET-2) on the elastic recovery and strain-in-compression of

127   experimental VPS and to compare it with commercial materials. In summary, addition silicone

128   materials with higher cross-link density have better elastic recovery, as they have a greater number

129   of cross-links that can resist deformation. This property, along with other desirable characteristics

130   such as tear strength and dimensional stability, make addition silicone impression materials a

131   popular choice for dental impressions. It was hypothesized that in the current study the

132   experimental formulations have better elastic recovery and strain-in compression-values due to

133   higher cross-links provided by adding TFDMSOS and Rhodasurf CET-2 and making it a more

134   suitable material for recording an accurate impression.

135

136   **Materials & Methods**

137   The ethical approval and informed consent were not required for this study, since this study do

138   not involve living human subjects and only involve *in vitro* laboratory testing of dental impression

139   material. Three medium-body commercial VPS impression materials were used in this study;

140   Aquasil Ultra Monophase, USA, Dentsply (Aq M), Elite HD Monophase, Italy, Zhermack (Elt M)

141   and Extrude, USA, Kerr (Extr M). Additionally, five experimental VPS formulations were

142   prepared as base paste and catalyst paste (Table 1) [5]. Exp-I was used as a control for Exp-II, while

143   Exp-II acted as a control group for Exp-III, Exp-IV and Exp-V.

144

145 **Preparation of Experimental Formulations**

146 The base paste of Exp-1 (hydrophobic VPS) was formulated by mixing vinyl-terminated poly-

147 dimethyl siloxane and a conventional cross-linking agent (poly methylhydrosiloxane) for 5

148 minutes using an electric hand mixer (Kenwood, kMix, UK). The filler (Aerosil R812S) was added

149 to the mixture and a uniform paste was made by mixing the components with a pestle and mortar

150 for 5 min, followed by blending the paste with an electric mixer for 10 minutes.

151

152 The catalyst paste was formulated by mixing vinyl-terminated poly (dimethylsiloxane), platinum

153 catalyst and palladium for 5 minutes with the electric hand mixer, followed by the addition of filler

154 (Aerosil R812S) and mixing it with the pestle and mortar and electric hand mixer. For preparation

155 of Exp-II impression material, the amount of poly (methylhydrosiloxane) was reduced from 1.10%

156 to 0.77% and it was replaced it with a novel cross-linking agent (TFDMSOS) in the base paste of

157 Exp-I formulation. Vinyl to silane groups were maintained at 1:1 ratio. The catalyst paste for Exp-

158 II was similar to that of Exp-I.

159 Experimental formulations III, IV and V were formulated by modifying base-paste of Exp-II with

160 addition of non-ionic surfactant; Rhodasurf CET-2) at concentrations of 2%, 2.5% and 3%

161 respectively. The quantities of constituents in the catalyst paste were adjusted to ensure adequate

162 polymerization of the materials (Table 1). The prepared base and catalyst paste of experimental

163 materials were kept in separate compartments of cartridge and stored at 4ºC ± 2ºC.

164 **Sample preparation for elastic recovery and strain under compression**

165 The cylindrical samples for elastic recovery and strain-in-compression were prepared using

166 polytetrafluoroethylene (PTFE) mould measuring 20 mm in length x 12.5 mm in diameter

167 according to ISO4823 (2007) standard. PTFE mould was positioned on top of a metal plate lined

168   with an acetate sheet. The base and catalyst pastes were syringed into the mould using an auto-

169   mixing syringe and the mould was sandwiched by another acetate-lined metal plate. The assembly

170   was held using C-clamp. Commercial materials were left to polymerize according to the

171   manufacturer's instructions while experimental materials were allowed to be set for 4 to 11

172   minutes (Din et al. 2017).

173   To measure elastic recovery (n=12), two metal plates ($13 \times 13 \times 3$ mm^3) were fixed on either side

174   of the specimen with the aid of double-sided sticky tape. The length of the specimen including

175   metal plates (h_1) was recorded using a digital micrometre (Mitutoyo, Japan) to an accuracy of

176   0.001mm. Then the specimen was deformed to 6 ± 0.1 mm within 1 second using the calibrated

177   mechanical testing machine (Tinius Olsen Ltd, Model H5KS, England, load cell 5kN) shown in

178   Figure 1. The deformation force was released slowly over a period of 5 seconds. After two

179   minutes the length was measured again (h_2). The elastic recovery in percentage, K, was assessed

180   using equation 1.

181

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

183   h_o is the height (mm) of the split mould

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

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

186

187   To evaluate strain-in-compression, 12 samples per material were tested. An initial force of

188   1.22 ± 0.1 N was exerted on the specimen and the distance (h_1) was calculated using the Tinius

189   Olsen (Figure 1). The load was increased to 12.25 ± 0.1 (N) progressively over a time of 10 seconds

190   at a rate of 3 mm/min and a change in height of the specimen was noted again (h_2). The percentage

191   of strain-in-compression, E, was calculated using equation 2.

192

193   **Equation 2: E** $= \left(\dfrac{h_1 - h_2}{h_0} \right) 100$

194   h_0 is the height (mm) of the split mould

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

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

197

198   The data was analyzed using SPSS Version 22 (Armonk NY IBM Corp, Armonk, NY, USA).

199   Numerical data were presented as mean and standard deviation. Analysis of variance was

200   performed with p value at 0.05. Where significant difference in group was found, individual

201   means were compared using post hoc Tukey's test.

202

203   **Results**

204   Table 2 shows the elastic recovery and strain-in-compression for commercial and experimental

205   VPS impression materials immediately after setting. All the tested materials met the ISO4823

206   requirement of having elastic recovery greater than 96.5%. Exp-II exhibited the highest elastic

207   recovery while Exp-V demonstrated the lowest values. The post-hoc analysis revealed that all

208   three commercial products and Exp-I had statistically similar elastic recoveries.

209

210   Use of novel crosslinking agent (TFDMSOS) instead of conventional agent significantly increased

211   elastic recovery. A significant difference in the elastic recovery was noted between Exp-II and

212   Exp-V. It was noted that the addition of a non-ionic surfactant (Rhodasurf CET-2) in the

213   experimental formulation, to improve hydrophilicity of material, resulted in a reduced percentage

214   of elastic recovery of material, however, the changes were statistically not significant (Table 2).

215

216   **Strain-in-compression**

217   Figure 2 and Table 2 reveal the strain-in-compression for the tested VPS impression materials.

218   Experimental VPS had significantly higher (p <0.05) strain-in-compressions values compared to

219   the commercial VPS. Exp-V exhibited significantly the highest (Tukey's HSD test) strain-in-

220   compression (7.08 % ± 0.22 %) while Elt M had the lowest values (3.15 % ± 0.18 %). Among

221   commercial materials, no significant difference between Aq M and Extr M was noted. However,

222   it was noted that the addition of a novel crosslinking agent i.e., TFDMSOS (Exp II), significantly

223   increased the percentage strain-in-compression values compared to formulations based on

224   conventional cross-linking agents (Exp-I, Aq M, Elt M, Extr M). Also, it was observed that

225   experimental formulations incorporating non-ionic surfactant (Rhodasurf CET-2) led to a further

226   significant increase in strain-in-compression values and this effect was concentration dependant.

227

228   Figure 3 and Table 2 show the comparison between elastic recovery and strain-in-compression for

229   all commercial and Experimental VPS impression materials evaluated in this study. Among the

230   experimental materials, there is a correlation between elastic recovery and strain-in-compression.

231   With the addition of TFDMSOS in Exp-II the elastic recovery and strain-in-compression increase

232   significantly compared to Exp-I (control). However, there is a negative correlation seen after

233   addition of Rhodasurf CET-2 (non-ionic surfactant) in Exp III. With the addition of surfactant the

234   elastic recovery is decreased while stain-in-compression is increased. It can also be noticed that

235   with the increase in % amount of surfactant there is a consistent and significant decrease in elastic

236 recovery and significant incurease in strain-in-compression in Exp-IV and Exp-V. Among

237 commercial materials, no significant difference was seen.

238

239 **Discussion**

240 The elastic recovery of the impression materials plays a major role in the accurate reproduction of

241 the oral cavity. The ability of elastomeric impression materials to revert to their actual form upon

242 removal of the applied stress is related to their coiled wrapped and kinked molecular chains. These

243 polymer chains stretch in the direction of stress and, recoil back on releasing the force, gaining

244 their original shape and form (Hamalian et al. 2011; Klooster et al. 1991). In the present study,

245 elastic recovery and strain-in-compression of commercial and experimental VPS impression

246 materials were compared.

247 Values for elastic recovery for commercial and experimental VPS impression materials ranged

248 from 99.11 to 99.64%. These values were within the range set by International Standards

249 Organization (ISO) 4823 (2007) which requires ≥96.5 %. Similar results were reported by Lawson

250 et al. (2008), who noted that elastic recovery of five tested VPS (Aquasil Ultra, Examix, Genie,

251 Imprint 3, and StandOut) and one hybrid impression material (Senn) ranges from 99.34 to 99.83

252 %. In another study Lu et al. (2004a) investigated the elastic recovery of two VPS (Flexitime and

253 Imprint II) and one polyether (Impregum). It was noted that Flexitime, Imprint II and Impregum

254 had 99.60, 99.75 and 99.19 % elastic recoveries respectively.

255 Similar percentage elastic recovery was noted for the commercial materials (Aq M: 99.32%, Elt

256 M: 99.31%, Extr M: 99.27%) and Exp-I (99.32%) containing conventional cross-linking agent

257 polymethylhydrosiloxane. However, on incorporating a novel cross-linking agent (TFDMSOS) in

258 Exp-II, an increase in elastic recovery (99.65%) was observed which was statistically significant.

259   The greater elastic recovery of Exp-II is attributed to excellent crosslinking of TFDMSOS with

260   functional groups of vinyl-terminated poly (dimethylsiloxane) pre-polymer as each molecule of

261   TFDMSOS can bond to four functional groups of pre-polymer (Din et al. 2018a). Similar results

262   have been reported in the literature indicating the amount of permeant deformation of an

263   impression material is greatly influenced by the degree of cross-linking of the polymeric chains

264   (Singer et al. 2022). The degree of polymerization also affects other mechanical properties of

265   elastomeric impression materials such as tear strength and % elongation-at-break (Din et al. 2018a;

266   Din et al. 2018b). It is reported that both the properties improved due to increased cross-linking of

267   polymeric chains.

268   The strain-in-compression was also calculated to assess the rigidity of impression materials so that

269   it can be removed from the mouth or cast without permanent deformation after setting, and to resist

270   deformation when the dental stone is poured. All tested impression materials have values for %

271   strain-in-compression within the ISO4823 (2007) limits. Experimental VPS impression materials

272   had higher strain-in-compression values indicating improved flexibility of experimental material

273   (Lu et al. 2004b). Therefore, a positive correlation between elastic recovery and strain-in-

274   compression was noted (Figure 3 and Table 2). Additionally, it was observed that the incorporation

275   of the wetting agent (Rhodasurf CET-2), further significantly increased strain-in-compression

276   values. This was contradictory to the results of Lu et al. who noticed that flexible materials have

277   less cross-linking and have better elastic recovery. This might be due to the difference in the

278   composition of the materials used in the present study.

279   The ability to undergo greater elastic recovery is a desirable property of impression materials as it

280   ensures an accurate impression which in turn guarantees a correct fit of the prosthesis. The

281   experimental VPS impression materials in this study show greater elastic recovery than their

282 commercial counterparts. Additionally, in previous studies, same experimental material has

283 proven to have improved wettability, percentage elongation, tear strength and minimal distortion

284 after disinfection making them a much more suitable option for impression taking (Din et al.

285 2018a; Din et al. 2018b; Din et al. 2021).

286 A limitation of this study is that it is conducted in an in-vitro environment under laboratory

287 conditions. To strengthen the claim of experimental VPS as a superior impression material, it is

288 necessary to conduct further research in intra-oral, in-vivo, conditions.

289

290 **Conclusions**

291 The addition of a novel cross-linking agent (TFDMSOS) showed improved elastic recovery and

292 strain-in-compression, while the addition of a non-ionic surfactant also significantly increased

293 strain-in-compressions values for all experimental VPS. All tested materials comply with ISO

294 standards. In the future, biocompatibility testing followed by clinical trials should be undertaken,

295 and material selection should be based on adequate knowledge of the properties of materials to

296 improve clinical success.

297
298
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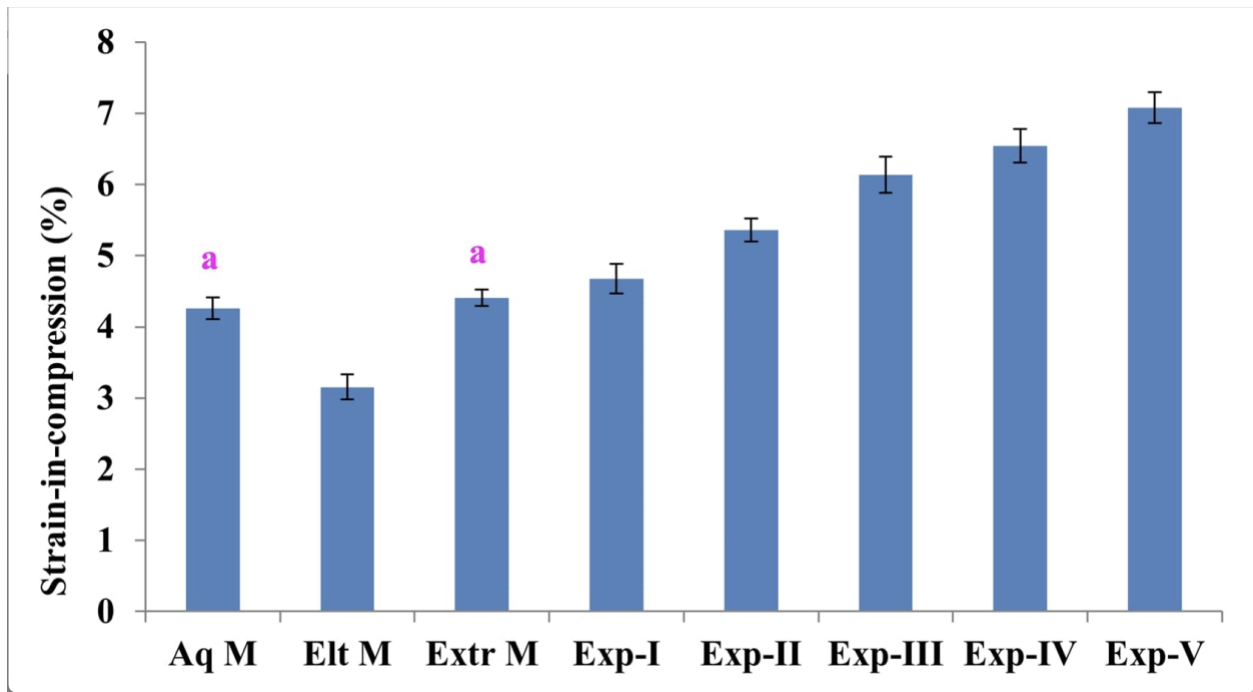357   *Materials Express* 8:85-92.
358
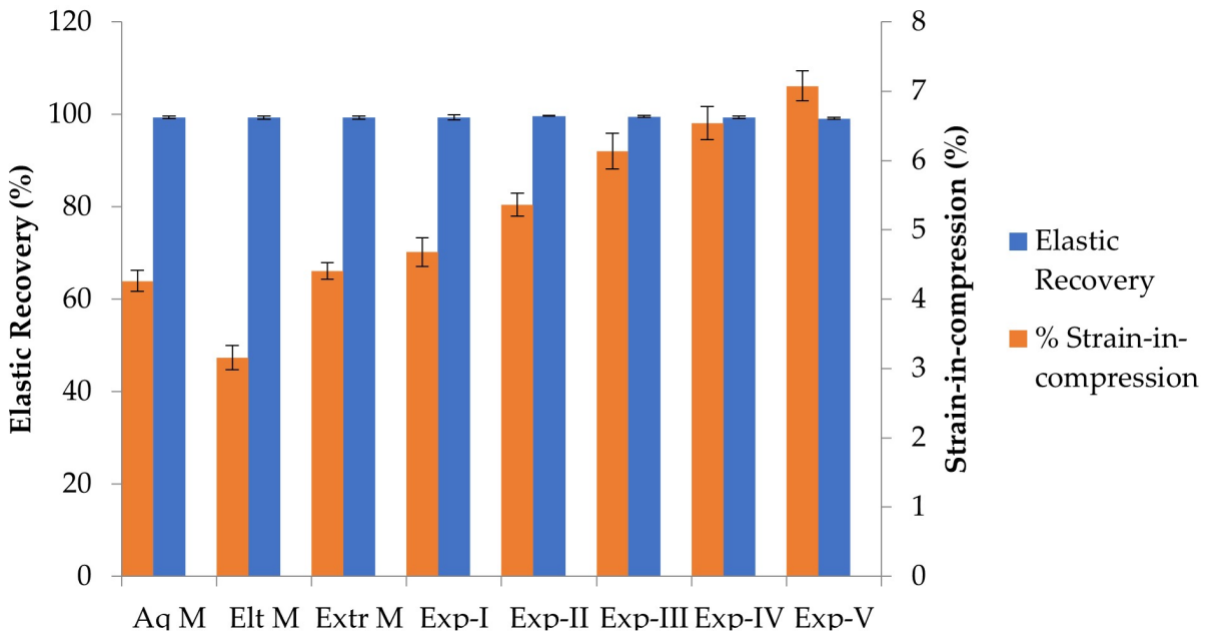
sample (20 mm length x 12.5 mm diameter)

Flat surfaces

Test specimen

Metal plates
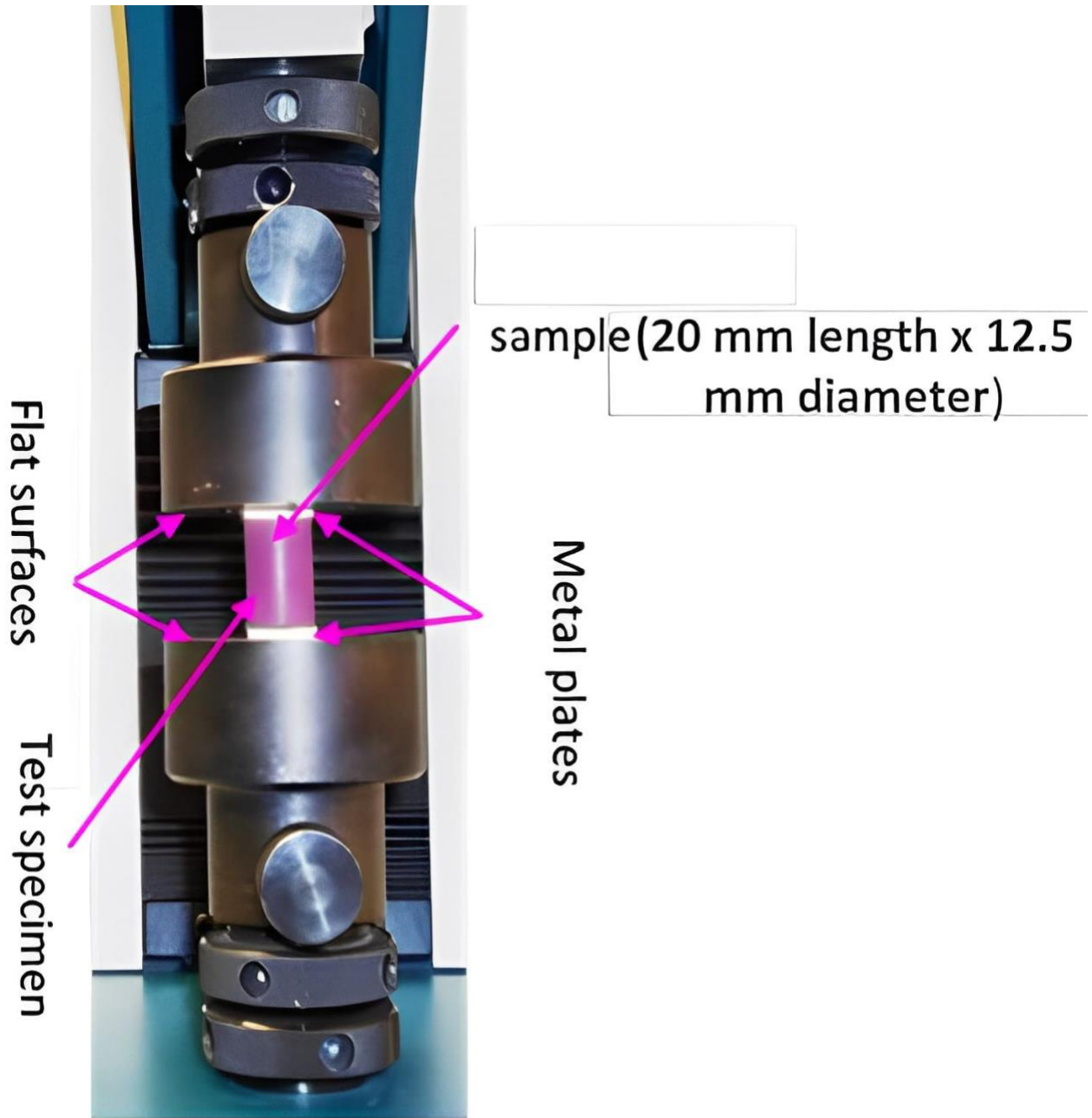
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360   **Figure 1: Tinius Olsen**

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363 **Figure 2: Strain in compression**

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366 **Figure 3: Comparison between elastic recovery and strain-in-compression**

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

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. 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