

1 **LOW ENERGY SPENT COFFEE GROUNDS BRIQUETTING WITH**
2 **ORGANIC BINDERS FOR BIOMASS FUEL MANUFACTURING**

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24 **ABSTRACT**

25 This paper analyzes the ability of the spent coffee grounds (SCG) for briquettes
26 production with xanthan and guar gums as binders. Briquettes were manufactured at room
27 temperature, at 15%, 20%, 25% and 30% of moisture, at 8 MPa, 10 MPa and 12 MPa of
28 compaction pressure and at 5 and 10% of binder dosage. Combination of 10% of xanthan,
29 15% of moisture, at 12 MPa, reached the highest dry density, 0.819 g/cm³. The
30 combination of 5% of xanthan, 30% of moisture, compacted at 12 MPa was the most
31 durable with a loss of mass of 3.9%. No relationships were established among water
32 absorption and binder type, binder dosage, manufacturing moisture or compaction
33 pressure. The lowest water absorption value, 0.25%, corresponded to the combination
34 with 5% of xanthan, 30% of moisture, compacted at 10 MPa. The lowest heating value
35 achieved by SCG was 25,399 J/g. Guar 5% and 10% combinations achieved 24,398 J/g
36 and 24,321 J/g respectively. Xanthan gum 5% and 10% dosages attained 24,450 and
37 23,503 J/g. Binder decreased volatiles, increased fixed carbon content and decreased
38 nitrogen content. Guar gum decreased SCG nitrogen content by 15.92% for the 5% and
39 by 16.92% for the 10% dosage combinations, respectively. Xanthan nitrogen reduction
40 attained 13.43% for the 5% and 14.43% for the 10% of dosage. The raw SCG ash
41 production was 0.66%. This value increased to 0.81 % and 0.97% with 5% and 10% of
42 xanthan gum, meanwhile guar decreased it to 0.57% and 0.52%, at 5% and 10% of
43 dosage.

44

45

46 **KEYWORDS**

47 Spent coffee grounds; biomass fuel; briquette; xanthan gum; guar gum.

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51 **1. INTRODUCTION**

52 Biomass is receiving an increasing interest as an effective and renewable source of energy
53 alternative to fossil fuels. Its use has demonstrated its ability to decrease the greenhouse
54 and acid gases emissions, the reduction of the energy production costs and saving
55 valuable and non-renewable resources such as petroleum or coal [1–4]. Despite its
56 potential, biomass shows two major disadvantages as sustainable source of green energy:
57 Nowadays most of biomass solid fuels are mainly produced from wood forest wastes
58 whose availability is limited. The worldwide increase of the energy needs could result in
59 the setting up of more and more energy crops which could compete with food crops for
60 arable lands. This could be supposed to be a big issue, mainly in developing countries
61 where biomass provides 20-33% of the total energy consumption. Agro-industry wastes
62 have demonstrated their ability for biomass fuel production [5–10]. Unprocessed biomass
63 bulk density and its usually small and irregular particle shape and size presents difficulty
64 in its storage, transportation, handling and feeding. This concern is usually overcome by
65 densification by high pressure and high temperature compaction processes. This way a
66 suitable commodity is produced with homogenous size and shape, improved density,
67 higher caloric density and lower emissions of particles and green-house gases, among
68 other improved properties [3,4,7,11,12]. However not all the agro-industry wastes have
69 the ability to become suitable solid fuel. One of the most usual limitations is the lack of
70 binding substances that produces densified biomass with insufficient mechanical strength.
71 In these cases it is required the use of organic or mineral binders to improve the final
72 product quality [8,13].

73 Spent Coffee Grounds (SCG) is a worldwide distributed agro-industry waste. The large
74 amounts of SCG generated every day, its pollutant potential, the lack of any effective
75 valorization way and its good lower heating power, make the SCG a high potential green
76 energy source Coffee is the most popular beverage in the world and the second traded
77 commodity [14], annual consumption of coffee bean is considered to be of 9.44 millions
78 of tonnes [15]. SCG has a high pollutant potential due to the toxic nature of substances
79 contained like caffeine, tannins, or polyphenols and requires high quantities of oxygen to
80 be degraded [9,16–18]. Seco et al. [19] demonstrated the low ability of this waste for its
81 densification and the potential of xanthan gum as binder for the SCG briquettes
82 manufacturing under low pressure and low temperature manufacturing conditions. This
83 paper continues the works carried out in the Public University of Navarre for the
84 valorization of the SCG as solid biomass fuel [19]. The aim of the investigation carried
85 out was to increase the nowadays available knowledge about the production of SCG
86 briquettes with organic binders. The effect of the use of xanthan and guar gums at two
87 dosages was investigated. Thus, the optimization of the manufacturing parameters could
88 allow a more effective valorization of large amounts of this waste, increasing the
89 sustainability of biomass solid fuels in accordance with the circular economy principles.

90

91 **2. MATERIALS AND METHODS**

92 One tonne of SCG was used for the laboratory investigation. The sample was
93 homogenized and physically- chemically characterized before testing. Details about this
94 sample and the xanthan gum used can be found in [19]. In this research, in addition to
95 xanthan, guar gum was used as binder. It is a water soluble polysaccharide polymer,
96 widely used as food thickener which is obtained from the *Cyamopsis tetragonoloba* seeds.

97 Every briquette was manufactured by filling a 65 mm diameter and 12 cm height
98 cylindrical mold, with 100g of material from every combination, being afterwards
99 compressed at 8 MPa, 10 MPa and 12 MPa. Once the compaction pressure was reached,
100 the samples were immediately unmolded. Manufacturing moisture contents were fixed at
101 15%, 20%, 25% and 30%. More details about the manufacturing methodology are
102 explained in [19]. Briquettes manufacturing parameters are shown in Table 1.

103

104

TABLE 1

105

106 Following the procedure of the previous paper, each combination was designated by the
107 code GT-MC-BI-DO-PR being:

108 GT the SCG type: raw (*R*)

109 MC the moisture content, followed by *W*

110 DO the dosage of the binder

111 BI the binder type: xanthan (*X*) or guar (*G*)

112 PR the compaction pressure

113

114 For example the code R15W5X8 identifies the combination containing raw SCG with
115 15% of wet content, dosage of 5% of xanthan gum and compaction pressure of 8 MPa.

116 Samples were maintained at room conditions (20° C and 50% RH) for a week before
117 testing. Dry density, durability and water absorption were stated for the characterization

118 of the quality of the briquettes. Dry density was considered representative of the ability
119 of biomass to be densified, durability test shows the resistance of the briquettes to

120 transport and handling and water absorption is an estimator of durability against
121 environmental conditions. Durability was characterized by means of an abrasion and

122 knocking test adapted from the European Standard UNE EN ISO 17831-2. Details about
123 the durability test carried out are shown in [19]. From a chemical point of view, proximate
124 and ultimate analysis as well as combustion tests were considered. Proximate analyses
125 were carried out in a METTLER-TOLEDO TG-DSC2 system. Tests were conducted with
126 10 mg of sample under an air flux of 100 ml/min and a heating rate of 10 C/min. A N₂
127 atmosphere was used from room temperature to 600° C and a N₂:O₂ (4:1) atmosphere
128 from 600° C to 900° C. Combustion tests were carried out in the same equipment and the
129 same conditions except the atmosphere, being a N₂:O₂ (4:1) oxidizing mix from room
130 temperature to 900° C. Elemental compositions of the raw materials were analyzed by
131 means of a ThermoFinnigan FlashEA 1112 analyzer. Tests were conducted in Helium
132 atmosphere at 900° C with oxygen injection and chromatographic columns gases
133 separation. Lower Heating Value (LHV) was determined using an IKA C5003
134 calorimeter.

135

136 **3. RESULTS AND DISCUSSION**

137 **3.1. DRY DENSITY**

138 Figure 1 shows the dry densities reached by all the combinations. The results obtained by
139 the raw SCG with no binder were added as reference.

140

141

FIGURE 1

142

143 For both binders and every moisture combination briquette dry density increased as
144 compaction pressure did. It was also observed an indirect relationship between the
145 moisture content and sample dry densities. This is due to the fact that water partially fills
146 the pores of the mixes, avoiding the occupation of these pores by the SCG particles during

147 compaction. Combinations of 10% of xanthan compared to those of 5%, showed an
148 increase of dry density for 15%, 20% and 25% of water content. For 30% of water content
149 a decrease of the sample densities was observed for 10% of xanthan gum dosage. The
150 combination R15W10X12 reached the highest dry density, 0.819 g/cm³, whereas the
151 lowest value, 0.672 g/cm³, was achieved by the combination R30W10X8. Guar gum
152 combinations reached lower density values than those containing xanthan gum for the
153 same binder dosage, moisture content and compaction pressure, showing lower
154 compaction ability. This binder achieved its highest dry density value for the combination
155 R15W5G12 with 0.768 g/cm³ and its lowest value for the combination R30W10G8 with
156 0.652 g/cm³, respectively.

157

158 **3.2. DURABILITY**

159 Figures 2-5 show the results obtained from briquettes containing both binders after the
160 durability test. Raw SCG samples were not tested because of the weakness of this
161 combination without binder whose samples broke up when handling.

162

163 FIGURE 2

164 FIGURE 3

165 FIGURE 4

166 FIGURE 5

167

168 Keeping constant the manufacturing parameters, durability test results improved as
169 moisture content increased for every dosages and compaction pressures. This
170 demonstrates the beneficial effect of using binders, the moisture content of the samples
171 and the compaction pressure, on the briquettes durability. Water acts as particle lubricant,

172 generates van der Waals' forces by increasing the area of contact between particles and
173 solubilizes the gums developing their binding ability [2,7]. 15% and 20% of moisture
174 content were insufficient to produce durable briquettes, getting the combination
175 R20W5X8 the best result, having lost 65.7% of the initial mass, at the end of the test.
176 Among the combinations with 5% of xanthan gum, the best durability results were
177 reached for the 30% of moisture content. Durability did not show any direct relationship
178 with the compaction pressure. The best result was achieved at 12 MPa of compaction
179 pressure, with a lost mass of 3.9% after 300 seconds test. At 8 MPa and 10 MPa, 4.0%
180 and 4.7% of lost mass were reached respectively. When the dosage of xanthan gum
181 increased up to 10% keeping constant the other manufacturing parameters, the mass
182 losses increased compared to 5%. For 10% of dosage the lowest loss of mass was
183 achieved by the combination R30W10X10, with 9.1%, followed by R30W10X12, with
184 21.9% and R30W10X12 with 22.5% of loss of mass respectively.

185 In the case of the guar gum combinations, a similar behavior to xanthan samples was
186 observed: durability increased as moisture content did, no relationship between energy
187 compaction and durability was observed and 5% of dosage reached better durability
188 values than 10%. The best durability result of the guar combinations was obtained by the
189 R30W5G12 samples, with a loss of mass of 19.8%. It is worth to notice that by comparing
190 combinations with the same parameters but with different binder, guar gum combinations
191 achieved worse results than xanthan gum ones did.

192

193 **3.3. WATER ABSORPTION**

194 Figures 6-9 show the results obtained by the briquette samples in the water absorption
195 test.

196

197

FIGURE 6

198

FIGURE 7

199

FIGURE 8

200

FIGURE 9

201

202 All the combinations showed an increase of absorbed water throughout testing time. No
203 relationships were observed among water absorption and binder type, binder dosage,
204 manufacturing moisture or compaction pressure. Among 5% of xanthan gum samples the
205 highest water absorption value was reached by the combination R15W5X8 samples with
206 1.08%. The lowest water absorption was attained by the combination R30W5X10, with
207 0.25%. For 10% of xanthan gum, water absorption oscillated between 1.10% and 0.32%
208 for the combinations R30W10X8 and R30W10X10, respectively. Among the samples
209 with 5% of guar gum the higher water absorption was achieved by the combination
210 R15W5G10, with 1.17% and the lowest by R15W5G12 with 0.59%. Finally, for 10% of
211 guar gum combinations, water absorption oscillated between 1.26% and 0.75% for
212 R20W10G12 and R30W10G8, respectively. Considering that water absorption values are
213 not higher than 1%, neither a significant trend can be observed in the different
214 combinations, nor significant differences between binders can be stated.

215

216 3.4. COMBUSTION CHARACTERIZATION

217 Table 2 shows the proximate and ultimate analyses of the different combinations as well
218 as their LHV.

219

TABLE 2

220

221

222 SCG showed adequate combustion characteristics compared to other biomass types,
223 specially its low ashes content [2,7,20–23]. It was found that the use of both binders
224 decreased the samples volatile content and increased the fixed carbon. Thus,
225 combinations with 5% and 10% of xanthan gum showed a decrease of volatiles content
226 to 76.35% and 75.85% meanwhile the combinations with 5% and 10% of guar gum
227 reached 76.43% and 76.33%, respectively. Considering the fixed carbon, combinations
228 with 5% and 10% of xanthan gum increased the SCG value from 18.00% to 19.01% and
229 19.05% respectively. Combinations with 5% and 10% of guar gum reached 19.07% and
230 19.20%, respectively. The decrease of volatiles and the increase of fixed carbon could
231 suggest a more stable combustion properties of the combinations containing binder
232 combinations compared to the raw SCG, slightly better for the guar than for the xanthan
233 gum. The ash content of the SCG, 0.66%, increased to 0.81 % and 0.97% with 5% and
234 10% for the xanthan gum samples respectively, pointing up a slight decrease of the quality
235 as solid fuel, but maintaining the ash content lower than 6% required by Standard ISO
236 17225-7 Standard for non-woody briquettes. Guar gum decreased the ash content to
237 0.57% and 0.52% with 5% and 10% of dosage, respectively. This suggest another
238 improved quality as fuel of the guar combinations as is their lower combustion wastes
239 generation. Ultimate analysis pointed up a decrease of the nitrogen content in the samples
240 treated with both binders which could suppose a potential decrease of the nitrogen based
241 compound emissions during combustion. This is more evident when guar gum is used as
242 binder, whose combinations with 5% and 10% of guar gum lowered the nitrogen content
243 by 15.95% and by 16.92% respectively, compared to the raw SCG.

244 Xanthan combinations N reduction achieved 13.43% with the 5% and 14.43% with 10%
245 of gum dosage respectively. None of the combinations tested showed S content, so S
246 compound emissions are not expected in the combustion of any combinations. Finally,
247 lower heating value of the raw SCG, 25,399 J/g, decreased with both binders, especially
248 for xanthan gum combinations. This is due to the partial substitution of higher energy
249 SCG compounds by these lower energetic value gums. As expected, LHV showed an
250 indirect relationship with binder dosage. Thus, guar 5% and 10% combinations achieved
251 24,398 J/g and 24,321 J/g, respectively. Xanthan gum 5% attained 24,450 J/g, reaching
252 the lowest value the combination containing 10% of xanthan gum, of 23,503 J/g.

253 Figure 10 shows the TG, DTA and DTG profiles of the mixes tested.

254

255

FIGURE 10

256

257 TG curves from all the combinations showed dewatering, volatiles combustion, fixed
258 carbon combustion and burnout stages. These curves suggest a lower content of volatiles
259 and fixed carbon of binder combinations compared to raw SCG in apparent contradiction
260 with the values showed in Table 2. This was attributed to the close results obtained from
261 each combinations, the instrumental errors and inhomogeneities of the small samples size
262 (about 10 mg). Xanthan gum DTA curves showed a decrease of the intensity of the
263 heatflow peaks corresponding to the fixed carbon compared to the SCG sample. It was
264 also observed a decrease of their temperatures from 515° C to the range of 500-505° C.
265 Compared to SCG, the intensity of the xanthan combinations volatile peaks increased,
266 appearing these peaks at lower temperatures, decreasing from 330° C to the range of 310°-
267 318° C. In the guar gum combinations the intensity of the fixed carbon peaks also

268 decreased and their temperatures changed to roughly 470° C. Guar combinations volatile
269 peaks intensity increased, and their temperature changed to roughly 310° C. For both
270 gums, peak intensity variations were directly related to their dosages. DTG curves showed
271 an increasing combustion rate for gum combinations in the range of the 260-380° C,
272 corresponding to the combustion of volatile compounds. These results suggest a lower
273 quality on combustion of the combinations containing gums and do not match with the
274 volatile contents shown in Table 2, by the same reasons than DTA curves. The highest
275 mass loss rate increase was reached by the 10% guar gum sample, followed by the 5%
276 xanthan, 5% guar and 10% xanthan. It was also noticeable the decrease of the temperature
277 of the secondary peaks compared to the raw SCG combination. It was also observed a
278 decrease of the rate peak temperatures, compared to SCG.

279

280 **4. CONCLUSIONS**

281 Xanthan and guar gums have demonstrated their ability for SCG briquettes manufacturing
282 by a low pressure and low temperature process. Meanwhile raw SCG briquettes were not
283 functional because of their lack of consistence, some of the samples containing gums
284 showed improved properties against handling, feeding and environmental conditions. The
285 gum type, dosage and moisture content demonstrated to be the most important
286 manufacturing parameters to manufacture briquettes with the required physical quality.
287 Thus, the best results were obtained with a 5% of xanthan gum and 30% of moisture
288 content. When combustion characteristics were analyzed, results were not conclusive: the
289 use of gums decreased the LHV of the SCG and nitrogen compound emissions. Xanthan
290 increased ashes generation meanwhile guar decreased it. Volatile or fixed carbon content
291 as well as combustion quality estimators got contradictory results which avoided state

292 clear combustion quality differences between the raw SCG and mixes where gum
293 additives were added.

294

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299

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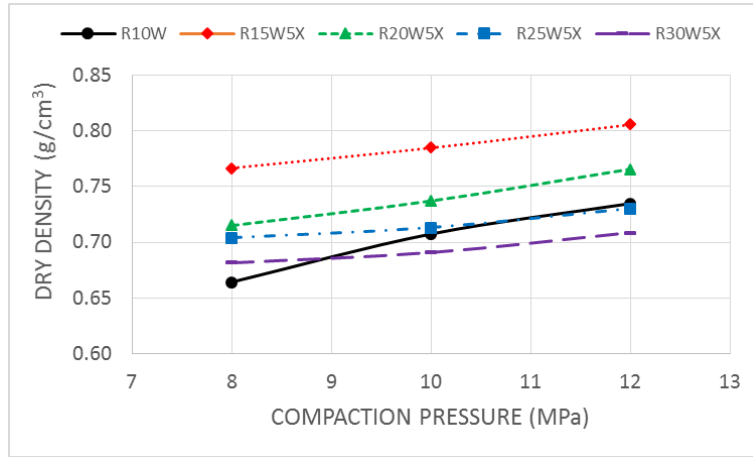
TABLE 1. Briquettes manufacturing parameters and sample codes.

SCG TYPE	MOISTURE CONTENT (%)	BINDER DOSAGE (%)	BINDER TYPE	PRESSURE (MPa)
RAW (R)	15	5	XANTHAN (X) GUAR (G)	8
	20			
	25			
	30			
	(W)			

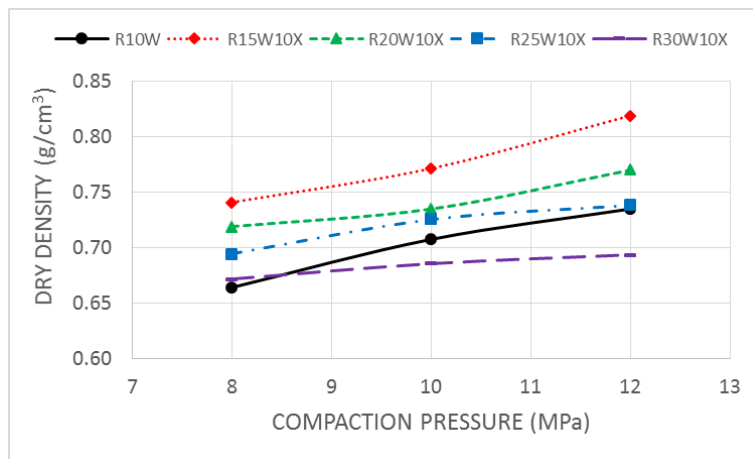
TABLE 2. Chemical characterization of the briquettes combinations and lower heating power.

Analysis	Raw SCG	SCG+5% of xanthan	SCG+10% of xanthan	SCG+5% of guar	SCG+10% of guar
Proximate analysis (% wt.)					
Moisture	2.46	3.84	4.14	3.93	3.96
Volatiles	78.88	76.35	75.85	76.43	76.33
Fixed carbon	18.00	19.01	19.05	19.07	19.20
Ash	0.66	0.81	0.97	0.57	0.52
Ultimate analysis (% wt.)					
N	2.01	1.74	1.72	1.69	1.67
C	57.29	57.23	57.20	55.79	54.50
H	7.52	7.78	7.84	7.48	7.37
S	0.00	0.00	0.00	0.00	0.00
O	33.18	33.25	33.24	35.04	36.46
Lower heating value (J/g)	25399	24450	23503	24398	24321

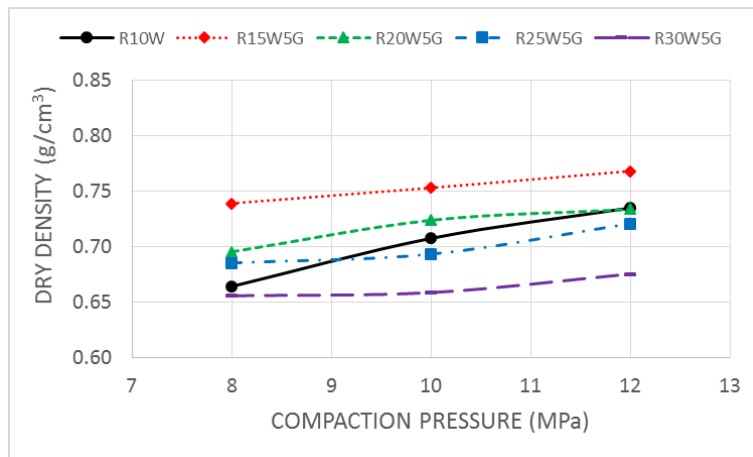
FIGURE 1. Dry density VS compaction pressure for different additives, dosages and moisture contents. a) 5% of xanthan gum, b) 10% of xanthan gum, c) 5% of guar gum and d) 10% of guar gum.



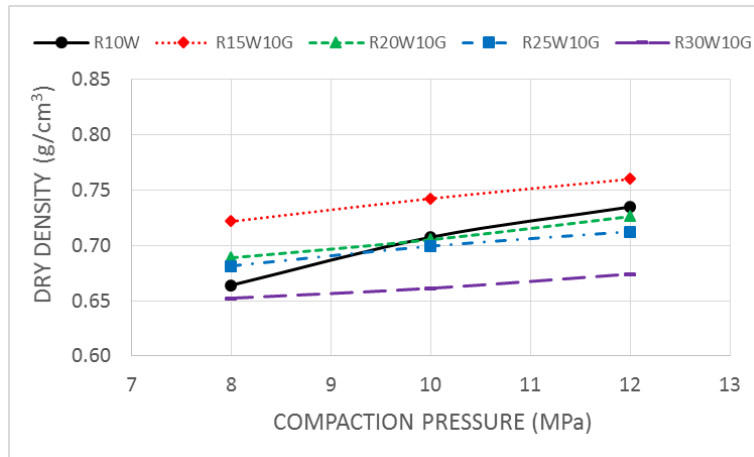
a)



b)

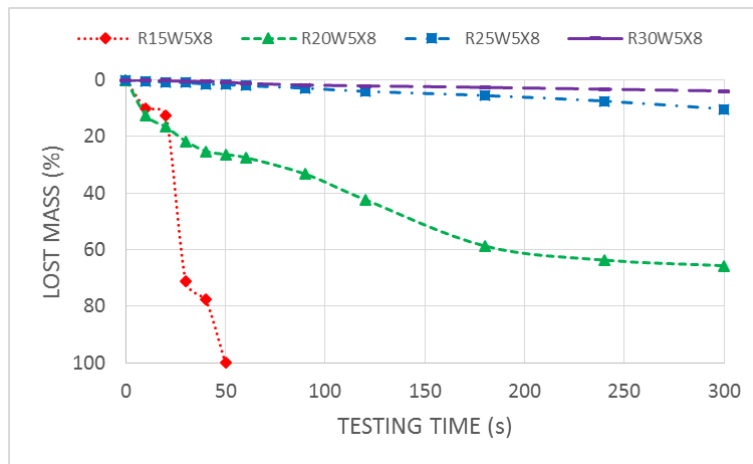


c)

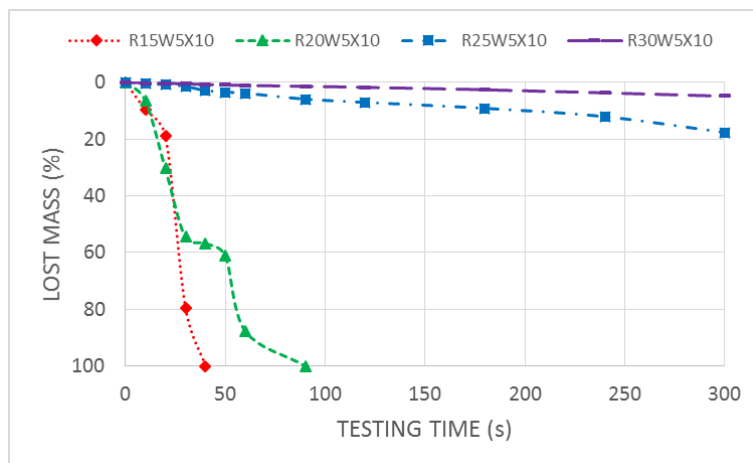


d)

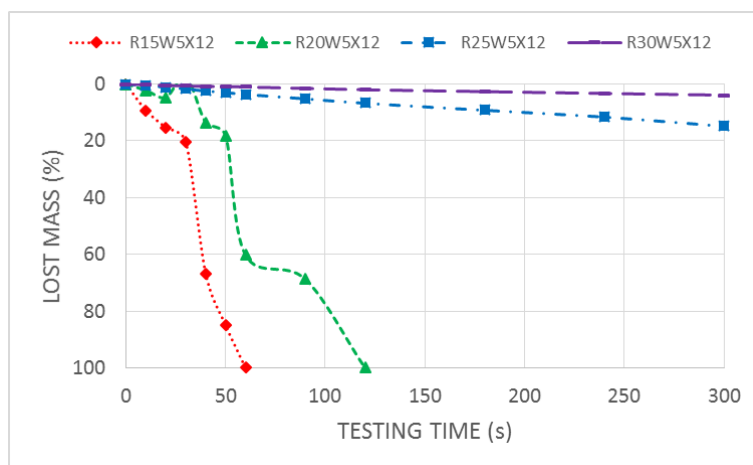
FIGURE 2. 5% Xanthan gum briquettes results for the durability test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

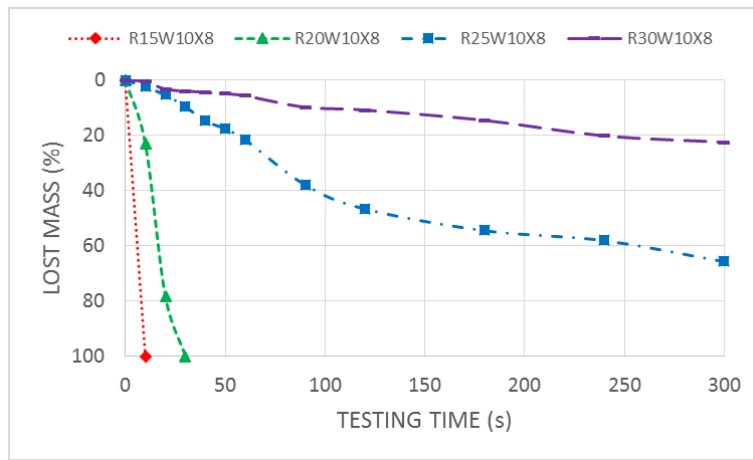


b)

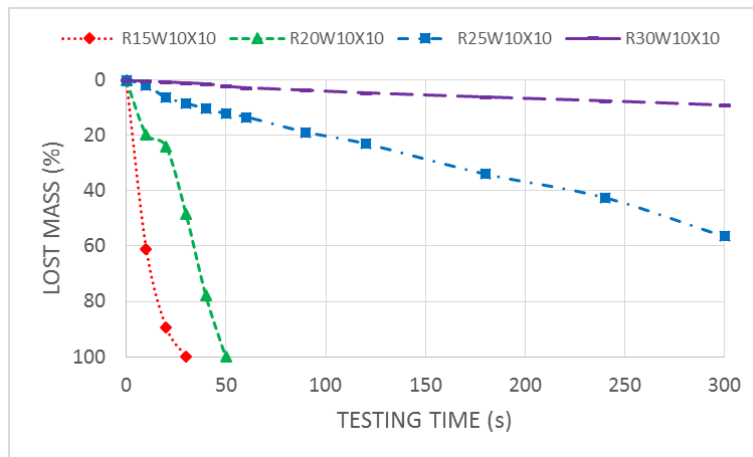


c)

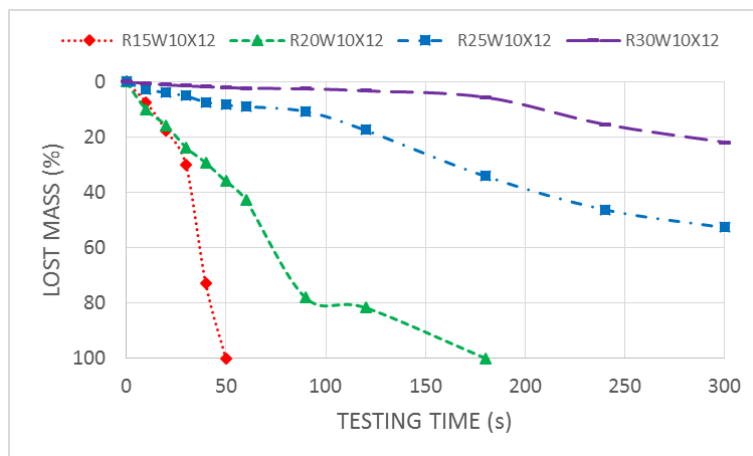
FIGURE 3. 10% Xanthan gum briquettes results for the durability test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

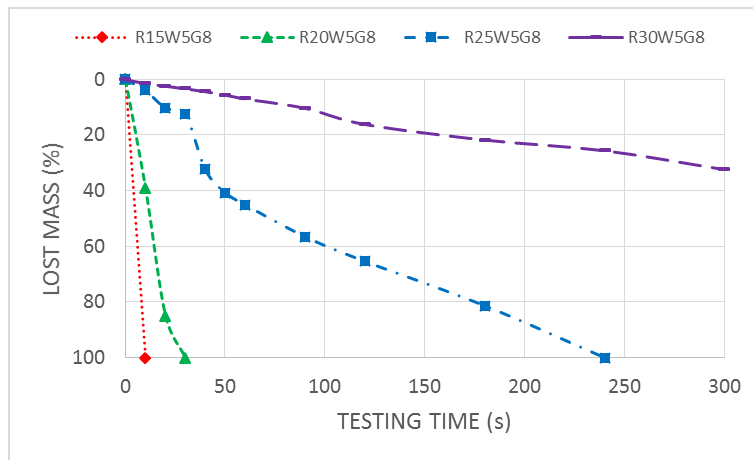


b)

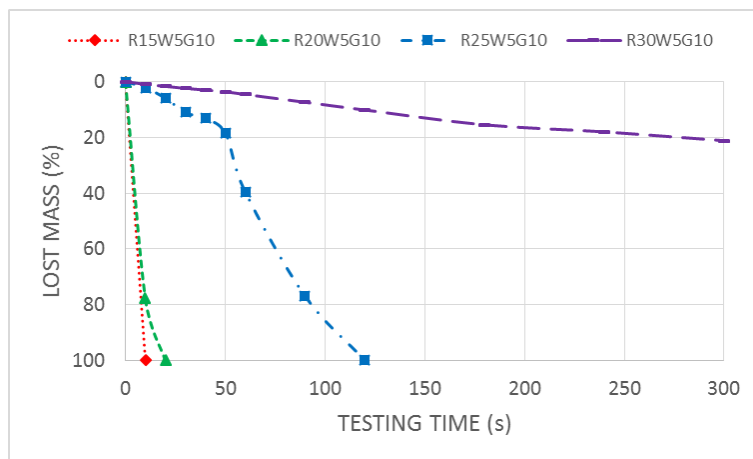


c)

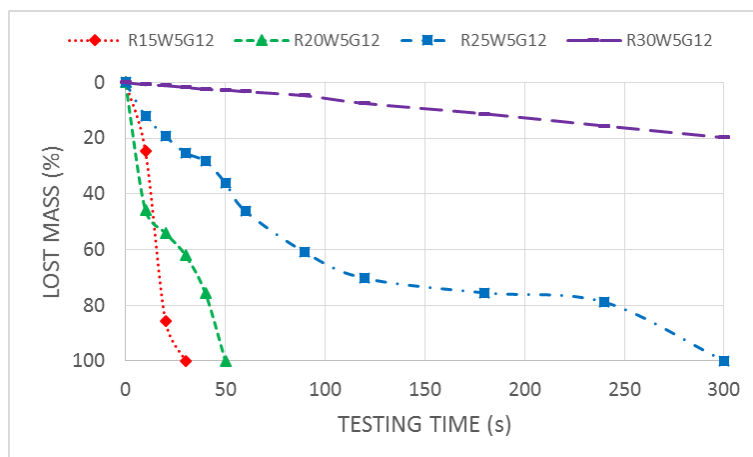
FIGURE 4. 5% Guar gum briquettes results for the durability test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

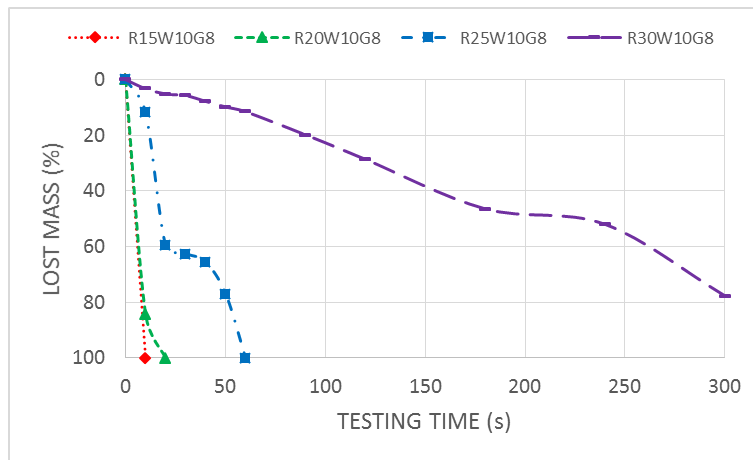


b)

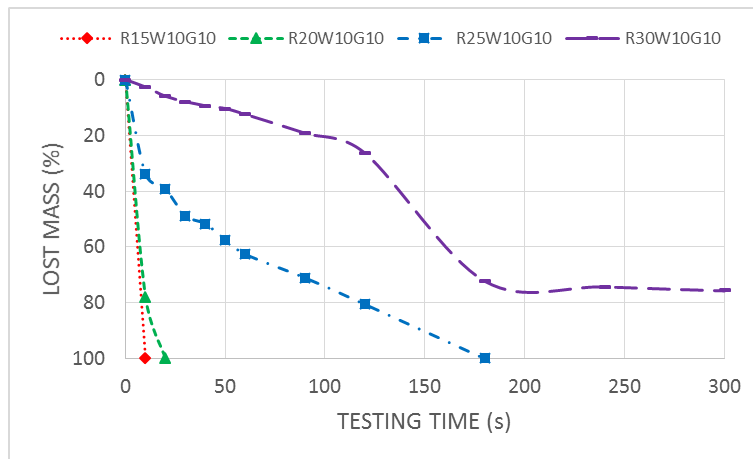


c)

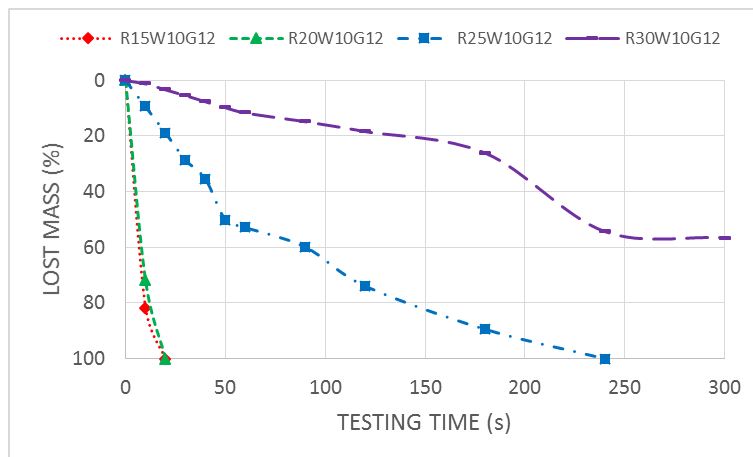
FIGURE 5. 10% Guar gum briquettes results for the durability test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

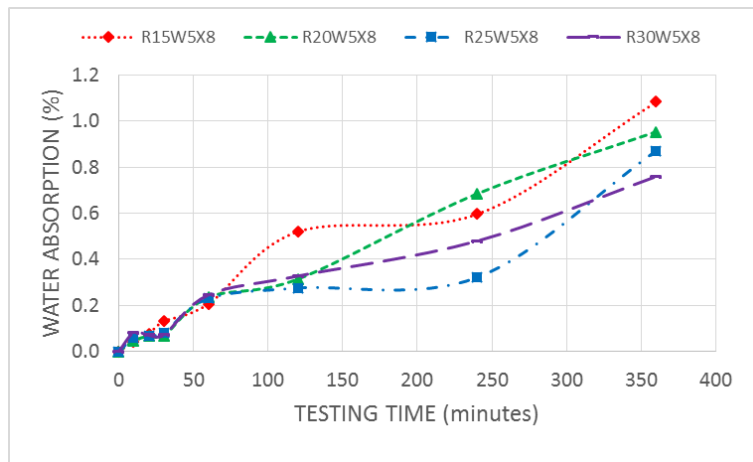


b)

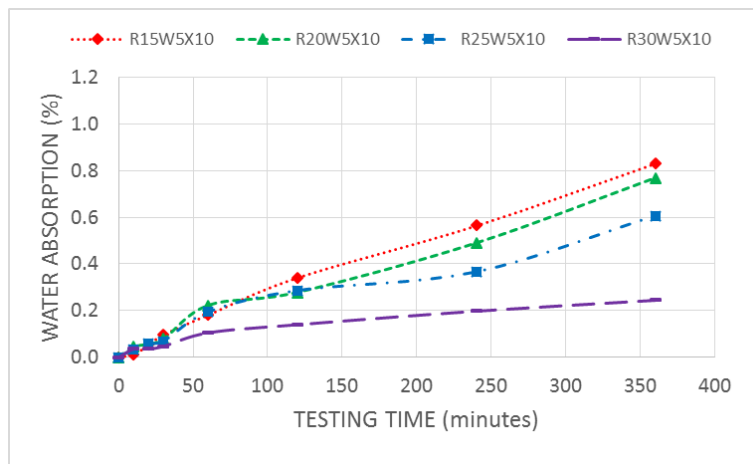


c)

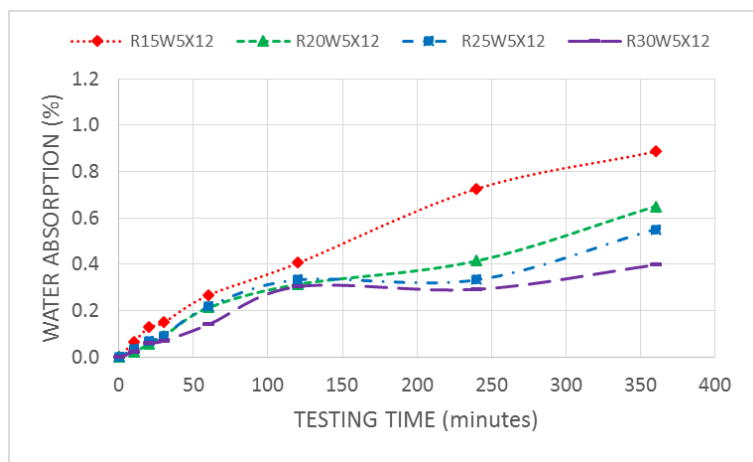
FIGURE 6. 5% Xanthan gum briquettes results for the water absorption test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

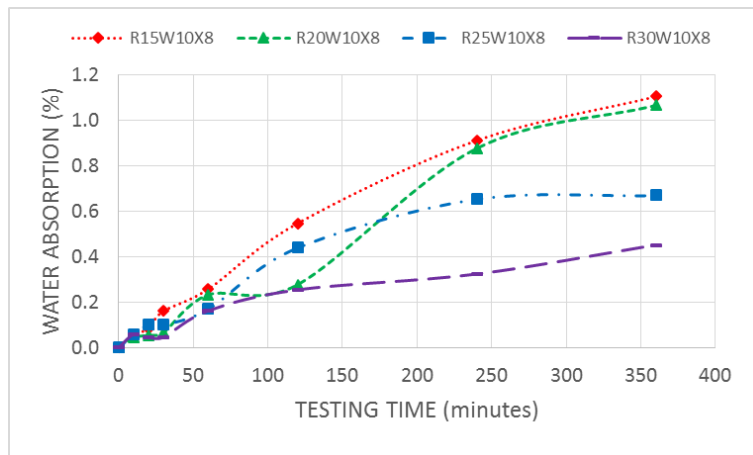


b)

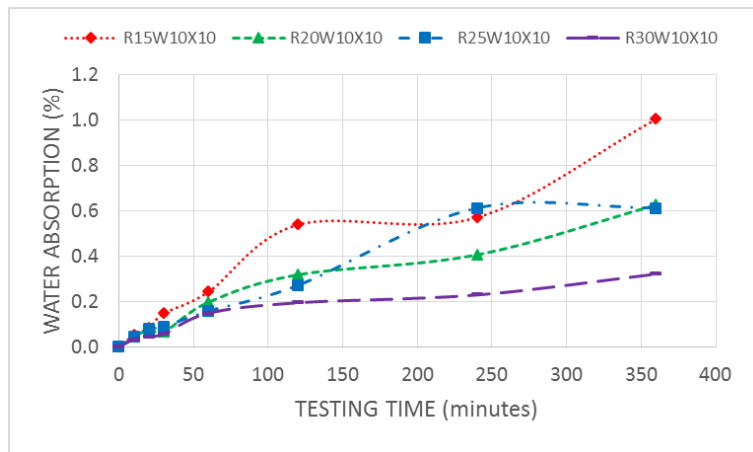


c)

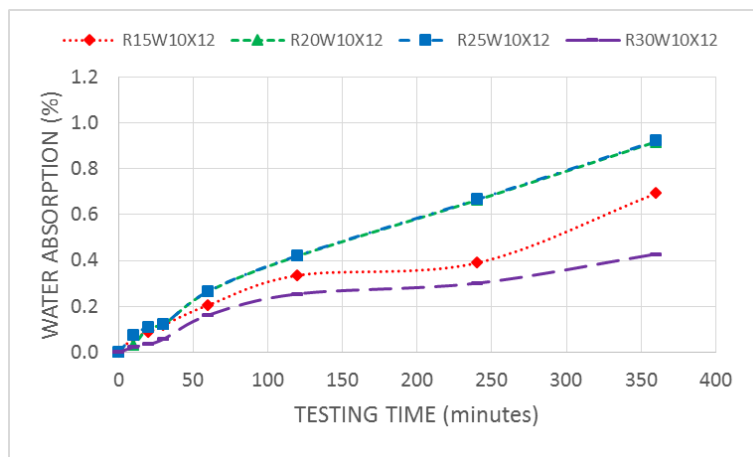
FIGURE 7. 10% Xanthan gum briquettes results for the water absorption test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

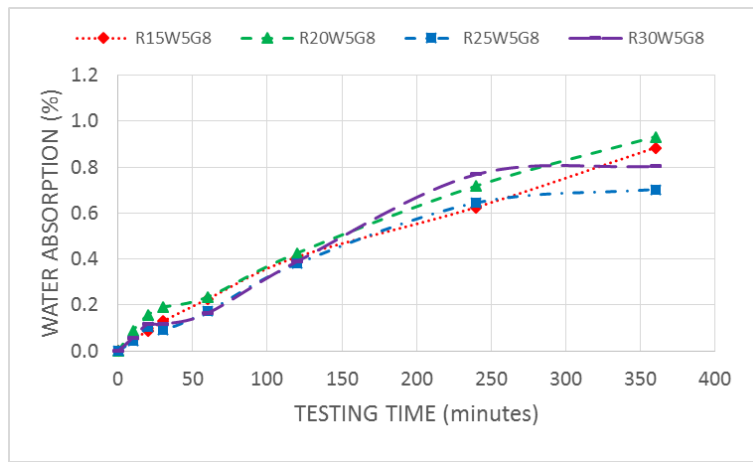


b)

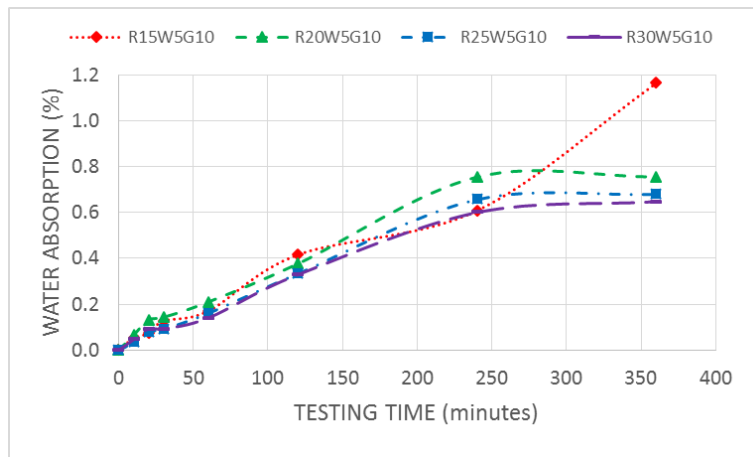


c)

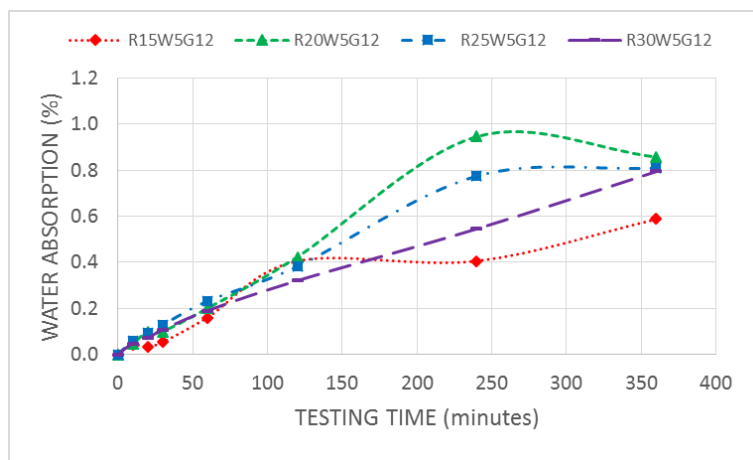
FIGURE 8. 5% Guar gum briquettes results for the water absorption test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

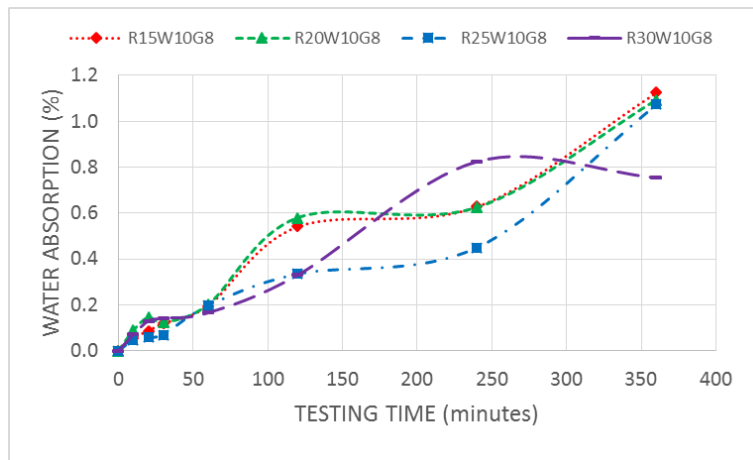


b)

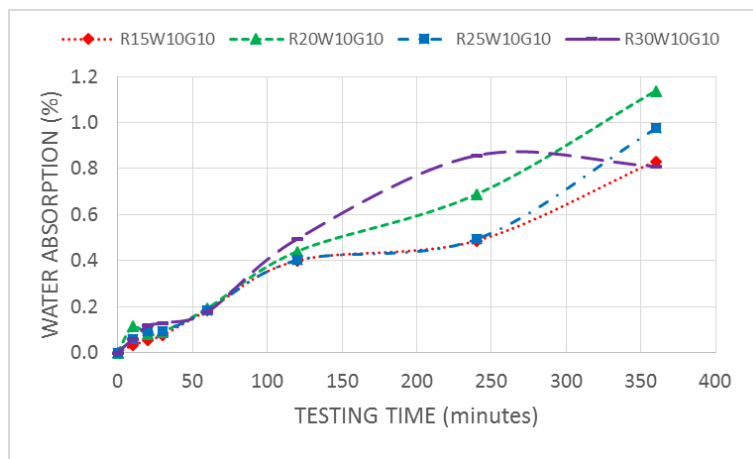


c)

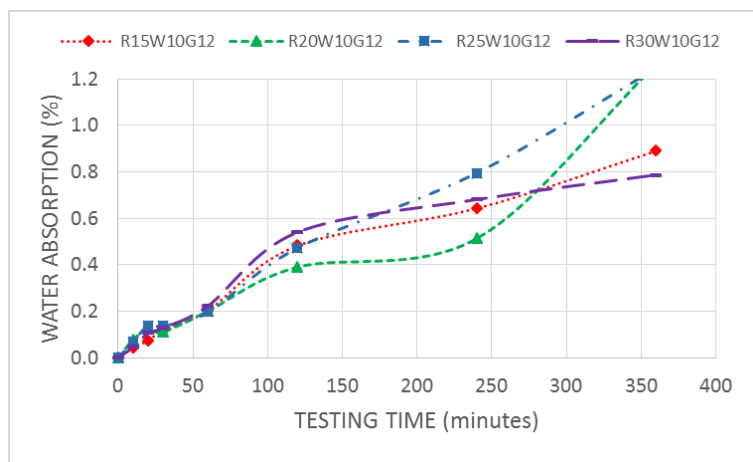
FIGURE 9. 10% Guar gum briquettes results for the water absorption test, manufactured with different compaction energies. a) 8 MPa, b) 10 MPa and c) 12 MPa.



a)

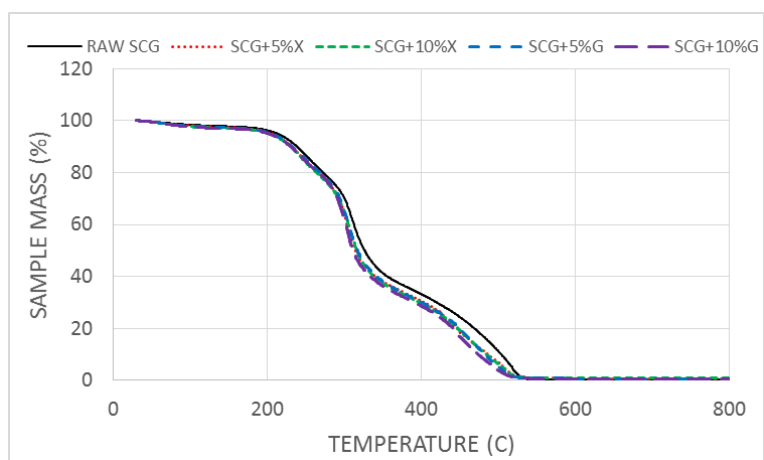


b)

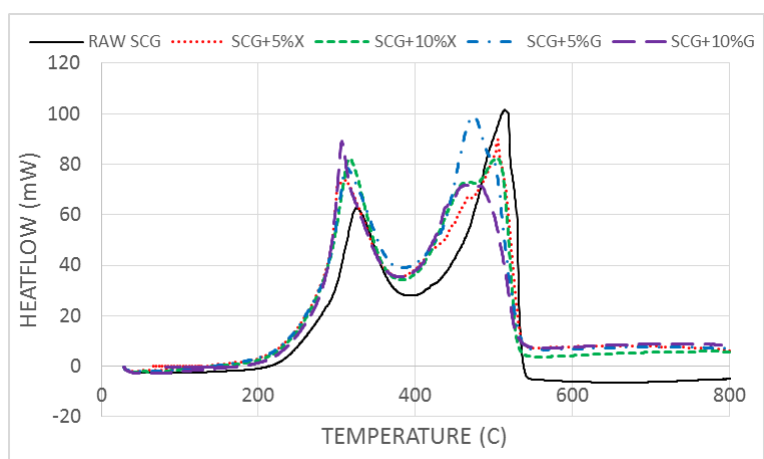


c)

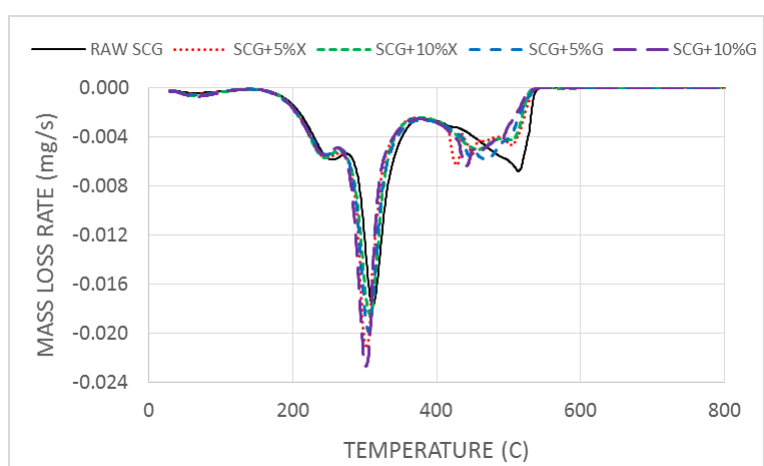
FIGURE 10. Raw SCG and xanthan and guar gum mixes results for the thermogravimetric analysis. a) TG, b) DTA and c) DTG.



a)



b)



c)