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Effect of storage on the physicochemical and nutritional properties of blueberry (*Vaccinium corymbosum* L.) jam

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Abbreviations

FRAP: ferric reducing antioxidant power, DPPH: 2,2-diphenyl-1-picrylhydrazyl, TPC: total phenolic content, S.D.: standard deviation, ANOVA: analysis of variance.

1 **Abstract**

2 This study investigated the effects of storage on the physicochemical and nutritional
3 aspects of blueberry jam. Jams were stored at either 4, 25, or 35 °C during a 56-day period.
4 The pH was significantly reduced during storage ($p<0.05$). Overall, results demonstrated
5 the significant effect of storage temperature and time on the color degradation and on the
6 texture of the samples studied ($p<0.05$). The total antioxidant activity was significantly
7 affected by temperature as the total antioxidant activity retention of samples stored at 4
8 °C was significantly higher to that of the samples stored at either 25 or 35 °C after a 56-
9 day storage period ($p<0.05$). A strong positive correlation was found between the total
10 phenolic content and the antioxidant activity with R^2 ranging from 0.617 to 0.716. Results
11 obtained herein suggested that blueberry jams should be refrigerated to better retain their
12 overall quality attributes and their antioxidant capacity.

13 **Practical applications**

14 Consumption of fresh blueberries may be limited because of seasonal and market
15 availability. Thermal processing strategies have been used in the food industry since
16 ancient times with the aim of not only making certain foods edible but delaying the
17 inevitable deterioration of perishable foods between production and consumption.
18 Blueberries and other berries can be available all year round in the form of jam. The
19 current study evaluated the effect of storage time and temperature on the physicochemical
20 and nutritional properties of blueberry jam. Results will add current knowledge to the
21 blueberry jam industry and facilitate the production of healthier blueberry jam.

22 **Keywords:** blueberry jam, thermal processing, storage temperature, antioxidant activity, jam
23 making, *Vaccinium corymbosum* L.

24 **1. Introduction**

25 Blueberries (*Vaccinium corymbosum* L.) are sweet, nutritious, and widely popular fruits
26 often labelled as superfoods because of their low calorie and high nutrient content.
27 Blueberries are rich in multiple antioxidants (Morita et al., 2017) and other
28 phytochemicals with biological properties such as anti-oxidant, anti-cancer, anti-
29 neurodegenerative, and anti-inflammatory activities (Seeram et al., 2006). Indeed,
30 previous studies carried out using high-fat fed rats suggested a potential anti-
31 inflammatory effect of blueberry supplementation associated with improved glucose
32 tolerance (Seeram et al., 2006).

33 Consumption of fresh blueberries may be limited because of seasonal and market
34 availability. Thermal processing strategies have been used in the food industry since
35 ancient times with the aim of not only making certain foods edible but delaying the
36 inevitable deterioration of perishable foods between production and consumption.
37 Blueberries and other berries can be available all year round in other forms such as nectar,
38 juice, canned, or jammed. This is achieved by the destruction of microbial pathogens and
39 the reduction of spoilage microorganisms as well as the inactivation of enzymes involved
40 in food deterioration. Jam making is one of the most popular fruit preservation methods
41 which can not only prolong the acceptability of fruits but also increase the availability of
42 any selected fruit during off-season (Rababah et al., 2011a). Jams are semi-solid gel-like
43 consistency food products that results from the cooking of the mixture of one or many
44 fruits, sugar, and water to achieve a concentration of total soluble solids higher than 68%
45 (Fügel et al., 2005). Other additives such as pectin or organic acids including citric or
46 acetic acid are generally used in jam formulations. Jam quality deteriorates from the time
47 it was produced until it is consumed (Ferreira et al., 2004). Previous studies suggested
48 that quality and shelf-life of jams depend on factors including processing (Poiana et al.,

49 2012), cultivar (Wicklund et al., 2005), or storage time and temperature (Touati et al.,
50 2014). For example, Touati et al. (2014) recently reported that the interaction time-
51 temperature had a significant effect on pH, total sugar and free amino acid content, and
52 the sensorial profile of apricot (*Prunus armeniaca*) jam during a 60-day storage period.
53 Furthermore, Patras et al. (2011) observed a decrease in the antioxidant activity of
54 strawberry jam after a 28-day storage period at either 4 or 15 ° C. In that study, results
55 showed a higher stability of nutritional parameters at 4 ° C when compared to 15 ° C.
56 The aim of this study was to assess the effects of different storage conditions during a 56-
57 day period on the physicochemical and nutritional properties of blueberry jam.
58 Physicochemical aspects studied included pH, color, and texture while the main
59 nutritional aspects evaluated were the total phenolic content (TPC) and the antioxidant
60 activity measured using the ferric reducing antioxidant power (FRAP) and the 2,2-
61 diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity assays.

62 **2. Materials and methods**

63 **2.1 Chemicals and reagents**

64 Methanol, DPPH, sodium carbonate, Folin-Ciocalteu's reagent, gallic acid, sodium
65 acetate, acetic acid, metaphosphoric acid, 2,4,6-Tris(2-pyridyl)-s-triazine, and ferric
66 chloride were purchased from Sigma-Aldrich (Steinheim, Germany). All reagents used
67 were of analytical grade. Blueberries, lemons, and sugar used for jam making were
68 purchased from local supermarkets.

69 **2.2 Homemade jam preparation and storage**

70 Blueberry jams were prepared following a traditional recipe. Briefly, blueberries were
71 washed thoroughly upon reception and each lot was divided into two equal parts: one was
72 mashed and the other one was left untreated. The juice of one lemon and sugar at a
73 blueberry to sugar ratio of 100:75 (w/w) were added to the blueberries and the mixture
74 was processed at 95 ° C during 20 min. After processing, 50 g of jam were poured into
75 sterilized 60 g glass bottles, left cool at room temperature, and sealed.
76 Jams were stored at either 4, 25, or 35 ° C during a 56-day period and were analyzed at
77 days 0, 7, 14, 21, 28, and 56.

78 **2.3 Color measurement**

79 Color recordings were taken using a Minolta CR-200 colorimeter (Minolta INC, Tokyo,
80 Japan). CIE values were recorded in terms of L* (lightness), a* (redness/greenness), and
81 b* (yellowness/blueness). Calibration was carried out using a standard white tile (Y:92.5,
82 x:0.3161, y:0.3321) provided by the manufacturer and the D65 illuminant, which
83 approximates to daylight.

84 **2.4 Texture**

85 Firmness was tested using a TA.XT-Plus Texture Analyser (Stable Micro Systems,
86 Surrey, UK) following the methodology described by Rababah et al. (2011a).

87 **2.5 Determination of the total phenolic content**

88 TPC was determined using the Folin-Ciocalteu's reagent following the method described
89 by Patras et al. (2011). Values measured at day 0 were used to calculate the decrease in
90 TPC, expressed as percentage decrease.

91 **2.6 Assessment of antioxidant activity**

92 Antioxidant activity was measured using two different methods: the FRAP and the DPPH
93 radical scavenging activity assay. The FRAP assay was carried out following the
94 methodology described by Wicklund et al. (2005). In addition, the DPPH radical
95 scavenging activity assay was carried out following the methodology previously reported
96 by Rababah et al. (2011a). Values measured at day 0 were used to calculate the decrease
97 in antioxidant activity, expressed as percentage decrease.

98 **2.7 Statistical analysis**

99 All tests were replicated three times. Results are expressed as mean \pm standard deviation
100 (S.D.). Difference between samples were analyzed using analysis of variance (ANOVA)
101 with JMP 13 (SAS Institute Inc., Cary, USA). Where significant differences were present,
102 a Tukey pairwise comparison of the means was conducted to identify where the sample
103 differences occurred. The criterion for statistical significance was $p < 0.05$.

104 3. Results and discussion

105 3.1 Physicochemical parameters

106 Prior to storage, the pH of the samples was 2.9 ± 0.0 , higher when compared to other jams
107 such as apricot (Touati et al., 2014), strawberry (*Fragaria ananassa*) (Rababah et al.,
108 2011b), or umbu-caja (*Spondias tuberosa*) (de Oliveira et al., 2015). The decrease in pH
109 was significant during storage ($p < 0.05$). Figure 1 shows the effects of storage time and
110 temperature on the pH. Overall, no differences were observed between the pH of the
111 different samples during the first 28 days of storage. However, after a prolonged storage
112 period at either 4, 25, or 35 °C the pH decreased from 2.94 ± 0.0 to 2.48 ± 0.01 , $2.40 \pm$
113 0.00 , and 2.40 ± 0.00 respectively. The decrease in the pH was higher after storage at 25
114 and 35 °C when compared to refrigerated storage at 4 °C ($p < 0.05$).

115 One of the most important parameters to which consumers are sensitive when selecting
116 foods is color. Table 1 lists the color attributes of blueberry jams during a 56-day storage
117 period. No differences were observed between the L^* and b^* values of jams stored at 4,
118 25, or 35 °C after 56 days of storage. However, Table 1 indicates that storage significantly
119 affected the color of the jams. The L^* value of jams stored at 4, 25, and 35 °C was slightly
120 higher after a 56-day storage period ($p < 0.05$). Results contrast with previous studies
121 which observed a decrease in L^* values after a 28 days of storage at 15 °C (Patras et al.,
122 2011). The opposite trend was observed for the a^* and b^* values where a significant
123 decrease was observed during storage ($p < 0.05$). The color intensity (chroma) was also
124 significantly reduced after storage from 1.2 ± 0.0 measured at day 0 to 1.1 ± 0.0 , $1.0 \pm$
125 0.0 , and 0.9 ± 0.0 after storage at 4, 25, or 35 °C, respectively ($p < 0.05$). Similar results
126 were observed previously in strawberry jams (Patras et al., 2011). Table 1 also shows the
127 gelling strength of the samples during storage. No significant differences were observed

128 between the firmness of samples stored at 25 °C at day 0 and after 56 days of storage.
129 However, during storage, the increase in firmness was significant for those samples stored
130 at 4 and 35°C (p<0.05). Storage also resulted in increased hardness in jam samples
131 previously (de Oliveira et al., 2015). Rababah et al. (2011a) showed no variations on
132 firmness of strawberry jams during a 15-day storage period at 45 °C.
133 Overall, results demonstrate the significant effect of storage temperature and time on the
134 color degradation and on the texture of the samples studied. New studies are needed for
135 sensory assessment during the storage of blueberry jam to evaluate the impact of
136 increased hardness on the sensory traits of the samples. Color differences could be caused
137 by the degradation of colored phytochemicals including anthocyanins that occurs during
138 thermal processing and storage. Indeed, Poiana et al. (2012) observed losses ranging
139 between 81 and 84% in the total anthocyanin content of bilberry (*Vaccinium myrtillus*)
140 jams during thermal processing. Similar results were obtained by Šavikin et al. (2009) in
141 black currant (*Ribes nigrum*) and black raspberry (*Rubus occidentalis*) jams.

142 **3.2 Nutritional properties: Total phenolic content and antioxidant activity**

143 Several research groups have suggested the importance of fruit phenolics as dietary
144 antioxidants. Compared to other fruits, berries including blueberries contain a high
145 antioxidant capacity, generally attributed to their high concentration of phenolics such as
146 anthocyanins (Moyer et al., 2002; Skrede et al., 2000). Figure 2 shows the effect of
147 storage time and temperature on the TPC of blueberry jams. Overall, there was a constant
148 decrease all throughout the storage time of the jams. Although no differences were
149 observed between the TPC of blueberry jams stored at either 4 or 25 °C at days 7 and 14,
150 the TPC of the samples stored at 35 °C was significantly lower when compared to the
151 initial TPC (p<0.05). Samples stored at 4, 25, or 35 °C during 56 days showed a decrease
152 in the initial TPC of 40.9 ± 1.6 , 32.5 ± 0.7 , and $25.4 \pm 1.7\%$, respectively. The observed

153 decrease was significantly higher in samples stored at 4 °C when compared to samples
154 stored at either 25 or 35 °C ($p<0.05$). Results suggested that the lower the storage
155 temperature, the higher the TPC loss. Results contrast with those obtained by Howard et
156 al. (2010) who reported that blueberry jams stored at 4 °C retained higher levels of
157 phenolic compounds including anthocyanins. In that study, a significant degradation of
158 anthocyanins was observed during storage. Similar results were obtained by Patras et al.
159 (2011) who suggested that during processing the cell structures are disrupted becoming
160 more prone to non-enzymatic oxidation, one of the main reasons for TPC loss. Therefore,
161 the observed reduction in the TPC during jam processing could be caused by a decrease
162 in total anthocyanins, the main polyphenols in blueberries (Rodriguez-Mateos et al.,
163 2014) or to disruptions in the cell structure during blueberry processing (Patras et al.,
164 2011).

165 The antioxidant activity was determined using both the FRAP and the DPPH radical
166 scavenging activity method and results are shown in Figure 3. Overall, results obtained
167 using the FRAP method suggested a constant decrease in the total antioxidant activity.
168 The observed decrease was significantly affected by temperature as the total antioxidant
169 activity retention of samples stored at 4 °C was higher to that of the samples stored at
170 either 25 or 35 °C after a 56-day storage period ($p<0.05$). The loss in antioxidant activity
171 assessed using the FRAP method was higher on samples stored at 35 °C when compared
172 to those stored at 25 °C during 56 days ($p<0.05$). Results obtained using the DPPH radical
173 scavenging activity were consistent with those obtained using the FRAP method.
174 However, no differences were observed between the antioxidant activity of samples
175 stored at 4 and 25 °C at day 56. Overall, results demonstrated that the antioxidant activity
176 of blueberry jams was significantly affected by storage temperature and duration. Similar
177 results were observed by Patras et al. (2011) who observed a 78.6 and 77.5% decrease in

178 the antioxidant capacity of strawberry jam after a 28-day storage period at 4 and 15°C
179 respectively. Amakura et al. (2000) reported a decrease on the antioxidant activity of
180 black currant, blackberry, raspberry, red currant, and strawberry jam by 50 to 60% of its
181 initial value. The antioxidant capacity is most significantly correlated with the contents
182 of total phenolics and anthocyanins (Poiana et al., 2012). In the current study, a strong
183 positive correlation was found between TPC and antioxidant activity with $R^2=0.617$ and
184 $R^2= 0.716$ for FRAP and DPPH, respectively. Similar results were found in strawberry
185 jam (Patras et al., 2011) and in orange (*Citrus sinensis*), cherry (*Prunus avium*), and fig
186 (*Ficus carica*) jams previously (Rababah et al., 2011b).

187 **4. Conclusions**

188 Berries and their derived products have often been proposed to possess health-promoting
189 properties. However, storage and processing can affect the nutritional content of these
190 products as well as their quality and overall acceptance. Therefore, the effects of storage
191 conditions on the quality attributes and on the nutritional properties of blueberry jams
192 should be considered prior to selection of storage conditions. Results obtained herein
193 suggested that blueberry jams should be refrigerated to better retain their overall quality
194 attributes and their antioxidant capacity. Further studies on the effect of storage time and
195 temperature on specific phytochemicals such as anthocyanins are needed.

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203 **Table 1. Colour and texture of blueberry jam during storage at either 4, 25, or 35 °C**

Time (days)	<i>L*</i>	<i>a*</i>	<i>b*</i>	<i>C</i>	Hue	Firmness (N)
4 °C						
0	25,02 ± 0,00 ^{Aa}	1,25 ± 0,00 ^{Ac}	0,17 ± 0,00 ^{Ac}	1,26 ± 0,00 ^{Ac}	7,74 ± 0,00 ^{Ab}	0,22 ± 0,00 ^{Aa}
7	25,78 ± 0,01 ^{Bc}	0,85 ± 0,16 ^{Ab}	0,07 ± 0,01 ^{Ab}	0,85 ± 0,16 ^{Aa}	5,08 ± 0,32 ^{Aa}	0,53 ± 0,06 ^{Bd}
14	25,83 ± 0,06 ^{Ac}	1,02 ± 0,09 ^{Bb}	0,04 ± 0,04 ^{Cb}	1,02 ± 0,09 ^{Aab}	2,64 ± 2,12 ^{Aa}	0,40 ± 0,04 ^{Bc}
21	26,85 ± 0,04 ^{Bd}	0,99 ± 0,02 ^{Bb}	-0,48 ± 0,04 ^{Aa}	1,10 ± 0,00 ^{Ab}	154,00 ± 1,50 ^{Bd}	0,56 ± 0,00 ^{Cd}
28	25,27 ± 0,13 ^{Bb}	0,01 ± 0,03 ^{Aa}	-0,53 ± 0,23 ^{Aa}	1,06 ± 0,13 ^{Aab}	82,73 ± 60,39 ^{Ac}	0,26 ± 0,00 ^{Bb}
56	25,33 ± 0,10 ^{Ab}	0,99 ± 0,04 ^{Bb}	-0,52 ± 0,08 ^{Aa}	1,11 ± 0,08 ^{Bab}	152,38 ± 2,01 ^{Bd}	0,44 ± 0,04 ^{Cc}
25 °C						
0	25,02 ± 0,00 ^{Aa}	1,25 ± 0,00 ^{Ad}	0,17 ± 0,00 ^{Ad}	1,26 ± 0,00 ^{Ac}	7,74 ± 0,00 ^{Aa}	0,22 ± 0,00 ^{Ab}
7	25,27 ± 0,55 ^{Ab}	0,96 ± 0,12 ^{Abc}	-0,06 ± 0,18 ^{Ac}	0,97 ± 0,11 ^{Aab}	85,81 ± 82,00 ^{Bbc}	0,19 ± 0,01 ^{Aa}
14	26,10 ± 0,07 ^{Bc}	0,82 ± 0,05 ^{Abc}	-0,34 ± 0,05 ^{Bb}	0,89 ± 0,03 ^{Aa}	157,24 ± 2,93 ^{Bc}	0,25 ± 0,08 ^{ABabc}
21	26,85 ± 0,04 ^{Bc}	0,99 ± 0,02 ^{Bc}	-0,48 ± 0,04 ^{Aa}	1,10 ± 0,00 ^{Ab}	154,00 ± 1,50 ^{Bc}	0,33 ± 0,02 ^{Bc}
28	25,27 ± 0,13 ^{Bb}	0,01 ± 0,02 ^{Aa}	-0,53 ± ± 0,22 ^{Aa}	1,06 ± 0,13 ^{Aab}	82,73 ± 60,39 ^{Ab}	0,17 ± 0,07 ^{Aab}
56	25,34 ± 0,03 ^{Ab}	0,73 ± 0,08 ^{Ab}	-0,75 ± 0,18 ^{Aa}	1,05 ± 0,07 ^{ABab}	134,57 ± 7,30 ^{Abc}	0,19 ± 0,02 ^{Aab}
35 °C						
0	25,02 ± 0,00 ^{Ab}	1,25 ± 0,00 ^{Ac}	0,17 ± 0,00 ^{Ac}	1,26 ± 0,00 ^{Abc}	7,74 ± 0,00 ^{Aa}	0,22 ± 0,00 ^{Ab}
7	24,92 ± 0,13 ^{Ab}	1,44 ± 0,26 ^{Bc}	0,16 ± 0,01 ^{Bc}	1,44 ± 0,26 ^{Bc}	6,51 ± 1,23 ^{Aa}	0,17 ± 0,03 ^{Aa}
14	26,63 ± 0,04 ^{Cd}	0,89 ± 0,13 ^{Ab}	-0,55 ± 0,00 ^{Ab}	1,04 ± 0,11 ^{Aab}	148,09 ± 2,60 ^{Bbc}	0,14 ± 0,04 ^{Aa}
21	25,05 ± 0,33 ^{Ab}	0,88 ± 0,01 ^{Ab}	-0,26 ± 0,47 ^{Ab}	0,96 ± 0,12 ^{Aa}	74,97 ± 70,43 ^{Ab}	0,17 ± 0,07 ^{Aab}
28	24,82 ± 0,11 ^{Aa}	0,79 ± 0,05 ^{Ba}	-0,43 ± 0,13 ^{Ab}	0,89 ± 0,11 ^{Aa}	151,36 ± 4,25 ^{Bc}	0,26 ± 0,03 ^{ABb}
56	25,56 ± 0,48 ^{Ac}	0,74 ± 0,03 ^{Aa}	-0,63 ± 0,04 ^{Aa}	0,97 ± 0,00 ^{Aa}	139,36 ± 1,88 ^{Ab}	0,34 ± 0,02 ^{Bc}

Different capital letters indicate significant differences between samples stored at different temperatures and different lower case letters indicate significant differences between values measured at different sampling points. The criterion for statistical significance was $p < 0.05$.

Figure captions

Figure 1. Effect of storage time and temperature on the pH of blueberry jams

Figure 2. Total phenolic content retention of blueberry jams during storage at different temperatures

Figure 3. Retention of the antioxidant activity assessed using the (A) FRAP and (B) DPPH· assays

Figure 1

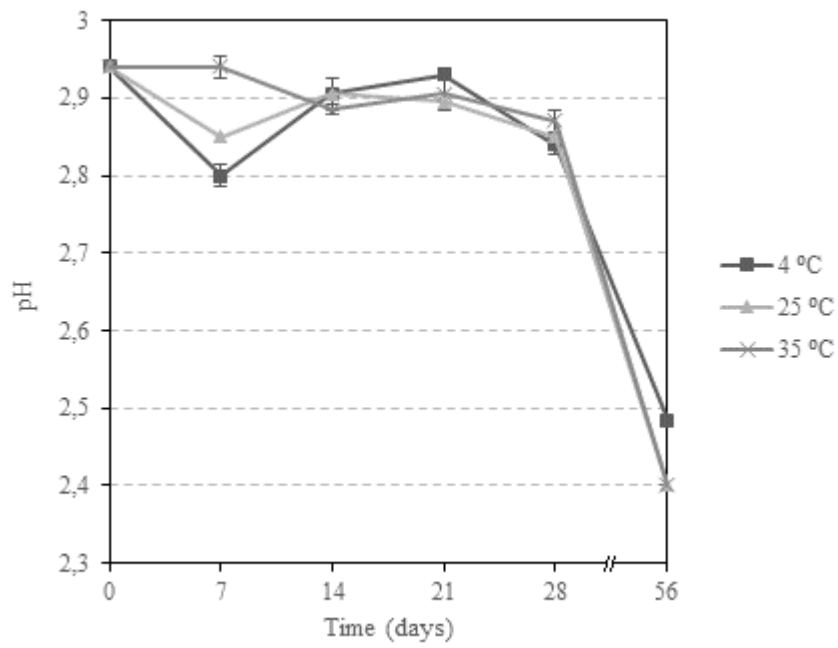


Figure 2

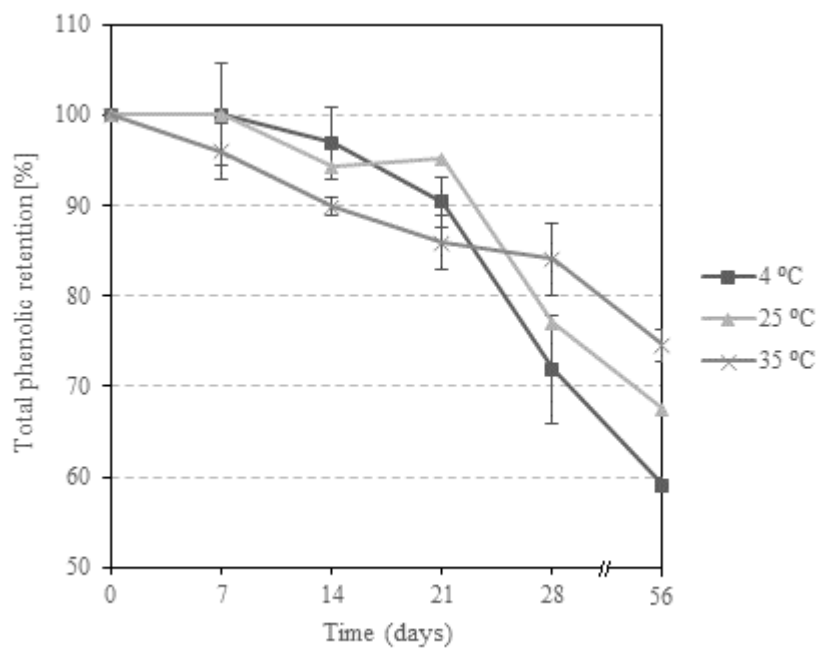
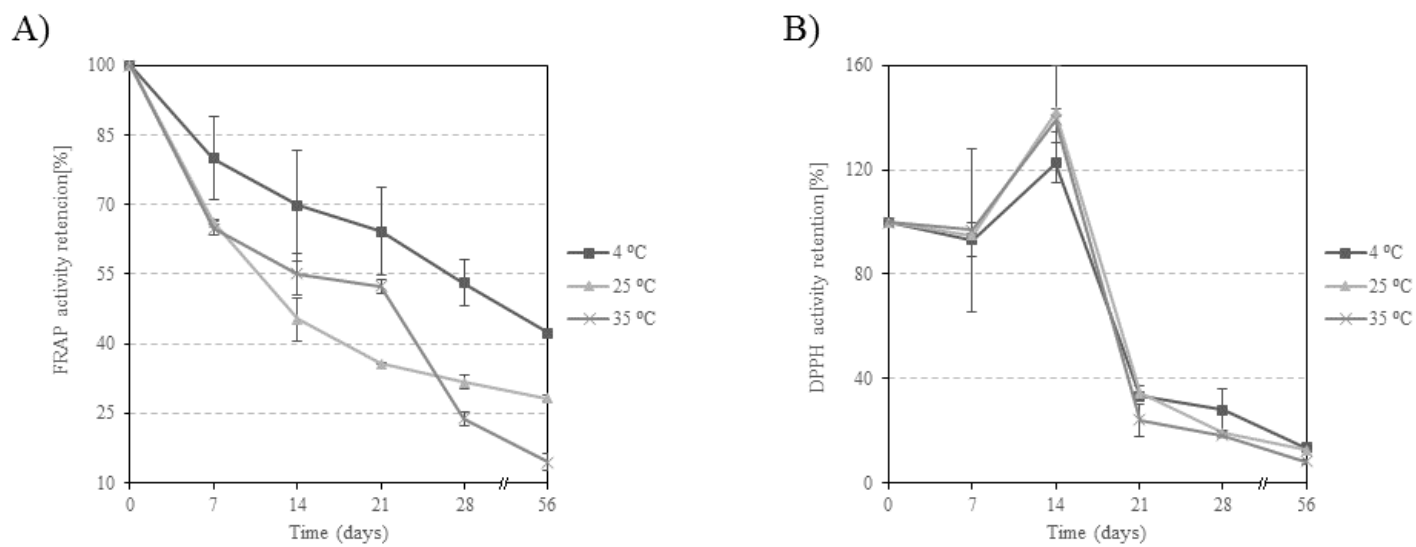


Figure 3



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