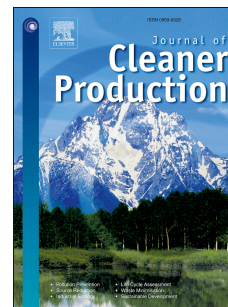


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Influence of waste glass in the foaming process of open cell porous ceramic as filtration media for industrial wastewater

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Title: Influence of waste glass in the foaming process of open cell porous ceramic as filtration media for industrial wastewater

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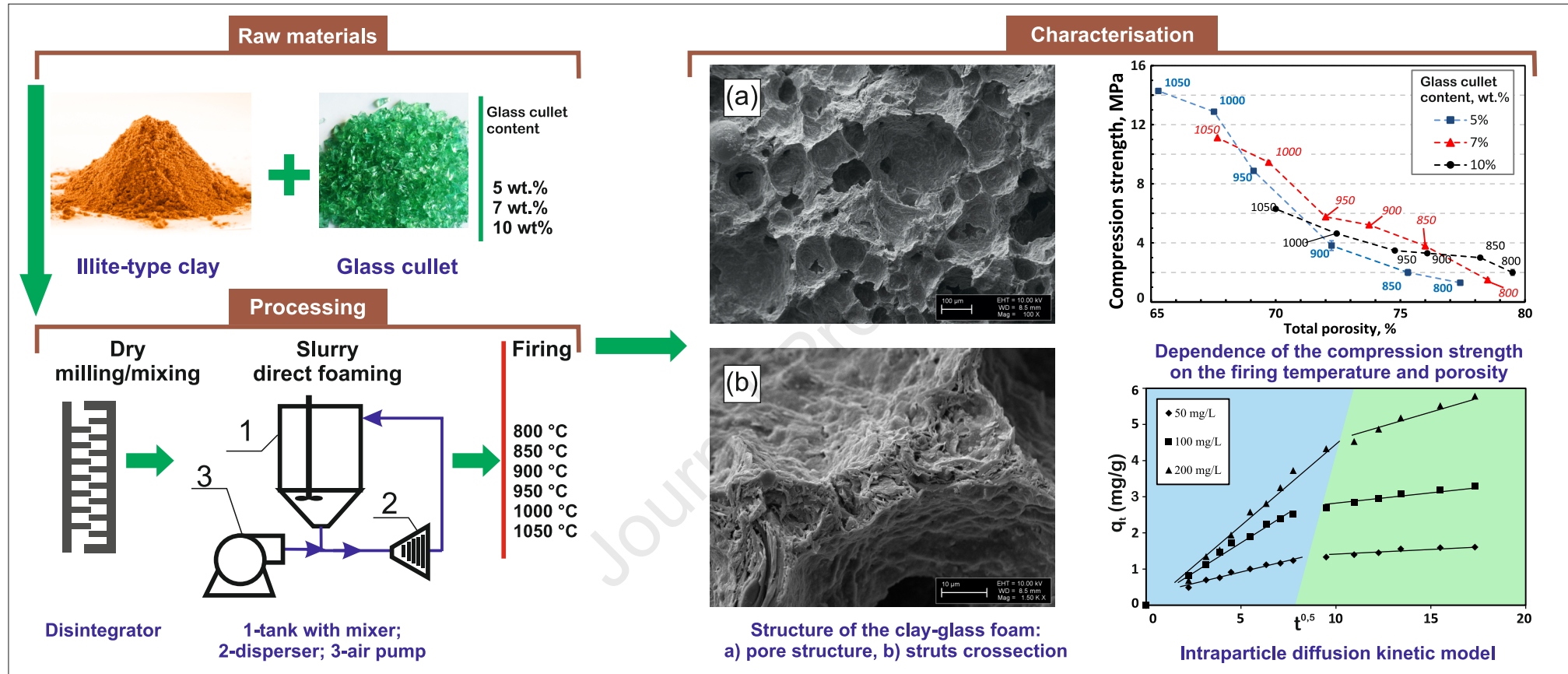
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Abstract

This paper reports the development and testing results of a prototype ceramic filter with excellent sorption properties (< 99% elimination in 5 min) leading to good efficacy in the removal of industrial contaminants (Reactive Bezaktiv Turquoise Blue V-G (BTB) dye). The novelty in the investigation lies in developing the filter material obtained from the recycling of waste glass combined with highly porous open-cell clay material. This newly developed material showed a significant reduction in the energy requirements (sintering temperature required for the production of industrial filters) thus addressing the grand challenge of sustainable and cleaner manufacturing. The methodology entails sintering of the clay foam (CF) at temperatures ranging from 800 to 1050 °C and blending it with 5%, 7% and 10 wt.% milled glass cullet. One of the aims of this investigation was to evaluate and analyse the effect of the pH of the solution, contact time and equilibrium isotherm on the sorption process and the mechanical compressive strength, porosity, water uptake. From the kinetic studies, it was

33 discovered that the experimental results were well aligned with the pseudo-second-order model and
34 chemisorption was discovered to be a mechanism driving the adsorption process. These findings are
35 crucial in designing cost-effective industrial filtration system since the filter material being proposed
36 in this work is reusable, recyclable and readily available in abundance. Overall, the pathway for the
37 reuse of waste glass shown by this work help address the sustainability targets set by the UN Charter
38 via SDG 6 and SDG 12.

39 **Keywords:** Ceramic clay foam, open cell, sorption capacity, textile dye, isotherm.

40 1. Introduction

41 The amount of waste glass, particularly soda-lime glass, is increasing year by year. In the
42 European Union, approximately 16.3 million tons of cullet was collected in 2016 (Eurostat,
43 2020). There is a pressing need to recycle waste glass and to address the theme of waste to
44 wealth. Accordingly, a number of studies have been conducted to use glass cullet as an
45 additional raw material or as a fluxing agent in ceramics (Ondruška et al., 2019) for instance,
46 as a surrogate to natural aggregates, in concrete to build pavements and roads (Majdinasab
47 and Yuan, 2019), and to obtain stoneware tiles and bricks (Chen et al., 2018; Lu et al., 2019;
48 Walczak et al., 2015). The use of cullet, particularly for municipally recycled glass, panel
49 glass, cathode ray tubes has been explored in greater details. Its usage has also been
50 investigated for the manufacturing of glass foams (Silva et al., 2018). Glass foams are
51 produced by combining various foaming agents. Primarily used foaming agents are aluminum
52 nitride, calcium carbonate, silicon carbide and manganese dioxide. The foaming process
53 energy requirement depends on the foaming agent: 700-725 °C for CaCO₃ (Bernardo and
54 Albertini, 2006) and 900-1000 °C for SiC (Francis et al., 2013).

55 In an earlier study glass-ceramics or porous components were manufactured using natural
56 materials and industrial waste, and a compression strength of ~ 80 MPa (Binhussain et al.,
57 2014) was achieved. It has been reported, that the addition of 10 wt.% of waste glass in clay
58 with firing at temperatures of 900-1000°C improved the mechanical traits (Phonphuak et al.,
59 2016) and reduced the firing temperature by 80-100 °C (Shishkin et al., 2020). However, very
60 few studies exist concerning addition of glass (Binhussain et al., 2014; Silva et al., 2018).
61 Taking into account clay-mineral diversity, there is extensive gap in the knowledge relating to
62 clay- cullet mix properties.

63 With the growing knowledge surrounding the ceramic material with the advantage of
64 their chemical stability, high porousness (Dong et al., 2012), high refractoriness, low mass

65 and thermal conductivity, as well as specific heat (Fukushima and Colombo, 2012), the
66 interest in these materials is increasing. Work has been done on different aspects such as
67 weight reduction (Goel et al., 2018), improved thermal and acoustic insulation properties
68 (Hostler et al., 2009), application as filters (Song et al., 2006) and catalyst support (Choo et
69 al., 2019). During the past decade, a wide assortment of approaches have been investigated
70 for producing absorbent ceramics, including the replica, the sacrificial template, water-oil
71 emulsion and the direct foaming methods (Colombo, 2008; Yue et al., 2011). However, only
72 one solitary study has been devoted to direct foaming technique despite it being fastest and
73 easiest method (Lakshmi et al., 2015).

74 Wastewater containing residual dye is produced primarily for textile and leather industries
75 (Yaseen and Scholz, 2019). The treatment technologies utilised at industrial level are either
76 based on activated carbon or Fenton's reagent based method (Wang et al., 2008). These
77 processes are expensive, particularly the regeneration of activated carbon is a tedious and
78 costly operation. In some cases, further treatments are required to purify the water (Durán-
79 Jiménez et al., 2014). So as to overcome the impediments of these technologies, this study
80 proposes porous ceramics as a material for water filtration. Reactive Bezaktiv Turquoise Blue
81 V-G (BTB) dye which is a synthetic anionic dye widely used in the textile industry, was
82 chosen as model organic pollutant. Adsorption of this dye by the designed product was
83 studied and related manufacturing conditions such as firing temperature and glass cullet –
84 Illitic clay mix ratio were analysed.

85 The overarching aim of this work was to produce clay foam (CF) with high adsorption and
86 adequate compressive strength. The methodology entails sintering of the CF at temperatures
87 ranging from 800 to 1050°C and blending it with 5%, 7% and 10 wt.% milled glass cullet.
88 The novelty in investigation lies in utilizing the waste glass combined with open cell porous
89 clay material that showed significant reduction in the sintering temperature (800°C). This
90 research considered various dye mixed water properties including pH (2-11), contact time (5-
91 300 min), and dye concentration (10-900 mg·L⁻¹). Optimum combinations of these factors
92 were determined to maximize the dye adsorption by clay foams, and the mechanisms involved
93 in the process were examined.

94 The rest of the paper is organised as follows: Section 2 provide details on fabrication
95 technique for clay foam along with batch experiments for dye adsorption. Section 3 details the
96 experimental results and relevant discussions. The paper concludes by highlighting that
97 sorption kinetic process follows the pseudo-second order kinetic model and chemisorption
98 was the dominant mechanism.

99 2. Materials and methods

100 Details of materials, fabrication methods, chemical and physical characterization techniques,
101 and batch adsorption experiment details are discussed below.

102

103 2.1. Reagents and Raw Materials

104 The illite type clay (deposition Liepa, Lode JSC, Latvia) was used for foam preparation.
105 This homogenised clay was secured from *Lode* Ltd brick factory (Latvia). The clay
106 agglomerate were dried at 105°C for 24 h, followed by refining in a jaw crusher to yield
107 particle sizes in the range of 10-20 mm. It was then subjected to milling using a laboratory
108 disintegrator DSL-175 (designed at Tallinn Technical University (Estonia) (Zimakov et al.,
109 2007)) for obtaining particle sizes < 50 µm. Green bottle glass was used as an milled cullet
110 (MC) source. Clean bottles were fed in jaw crusher, in order to obtain particle size of 10-20
111 mm. Subsequently they were also milled using the DSL-175 to yield particle sizes < 50 µm.
112 Detailed analysis of chemical and mineralogical composition of the clay and cullet used here
113 can be referenced from our previous work (Shishkin et al., 2020) but some salient details are
114 provided here. Oxide composition of the used clay are: SiO₂ - 62.8 wt%, Al₂O₃ - 15.4 wt%,
115 Fe₂O₃ - 6.8 wt%, CaO - 0.7 wt%, MgO - 1.4 wt%, Na₂O - 0.1 wt%, K₂O - 4.2 wt% TiO₂ -
116 1.9 wt%; And for MC SiO₂ - 70.2 wt%, Al₂O₃ - 2.1 wt%, Fe₂O₃ - 0.1 wt%, CaO - 9.5 wt%,
117 Na₂O - 16.6 wt%, Fatty alcohol sulfate preparation - Schäumungsmittel W53 Flüssig
118 (Zschimmer & Schwarz GmbH & Co KG Chemische Fabriken, Germany) was used as
119 foaming agent (FA). Municipal utility tap water (Riga, Latvia) and deflocculation agent made
120 of humates and silicates basis Dolaflox B11 (Zschimmer & Schwarz GmbH & Co KG
121 Chemische Fabriken, Germany) were also used for the clay slurry preparation. Phase
122 composition after adding glass was as follows: (i) 800 °C- Quartz, Illite, Haematite,
123 Microcline, Diopside, and amorphous phase (ii) 900 °C- Quartz, Illite, Haematite, Microcline,
124 Diopside, Spinel, Cristobalite, and amorphous phase.

125

126 2.2. Mixing and milling

127 The dry clay and cullet was milled and homogenised using the laboratory disintegrator
128 DSL-175 at a speed of 12000 rpm. Clay foam (CF) was obtained by direct foaming using
129 similar process as reported in previous study (Shishkin et al., 2015) utilising high-speed
130 mixer-disperser device (HSMD) (Corvus Ltd., Riga, Latvia). The core idea of the HSMD is
131 focused on ceramic slurry treatment producing multiple impacts in a liquid medium

132 (cavitation effect) by dispersing elements through the intensive homogenisation thus
133 introducing micro (50-100 μ m) bubbles (direct foaming) in bulk product.

134

135 2.3. CF Production Method

136 The mix of clay with MC content of 5%, 7%, and 10 wt.% was prepared as the first step.
137 Preparation of the CF comprised of the following stages. The HSMD was operated in
138 circulation mode and set at 500 rpm. It was filled with 600 ml of water and 1 wt.% of
139 dispersant (calculated from dry clay-glass mixture – 14 g). A total of 1400 g dry clay-MC mix
140 was slowly (~ 300 g.min⁻¹) added to prevent agglomeration and the HSMD speed was
141 gradually increased to 4000 rpm. The 5.5 wt.% of foaming agent (calculated from dry clay-
142 glass mixture – 77 g) was added after 30s and the mixer speed was gradually raised to 6000
143 rpm. With air introduction into the suspension, the foamed mix volume increased twofold.
144 Treatment by HSMD was continued in recirculation mode (1 min). The CF was then fed into
145 a mould of size 150x150x60 mm and naturally dried for 72 hours at room temperature. The
146 sample was additionally dried at 105°C for 24h. Oven dried sample were removed from the
147 mould, cut into 55x55x110 mm specimens and fired in a muffle furnace (LH11, P330 by
148 Nabertherm) at 900, 950, 1000 and 1050 °C at a rate of 5 °C min⁻¹ with a dwell time 30 min.
149 The sintered CF samples were cut and polished to a final size of 50x50x50 mm for further
150 tests.

151

152 2.4. Determination of the Physical and Mechanical Properties

153 Firing shrinkage was evaluated by direct sizing of the sample's linear dimensions
154 (55x55x110 mm) before and after firing. The water absorption, apparent density, bulk density
155 and apparent porosity were analysed by the pycnometry and Archimedes method (*Annual*
156 *book of ASTM standards, ASTM Standards C20, 2015*). Compressive strength of the sintered
157 specimens (25x25x25mm) were assessed using a Universal Testing Machine (UTM) (Instron
158 8801, Germany) in accordance with ASTM D695. All experiments were organized based on a
159 set of six samples for each category (glass addition and firing temperature) and results
160 reported.

161 For the microstructural characterization, optical microscope (Keyence Corporation, Osaka,
162 Japan, VHX-2000 and VH-Z20R/W lens) was used. Scanning electron microscope (SEM)
163 Zeiss EVO MA-15 (Carl Zeiss AG, Oberkochen, Germany) was used for microstructural
164 characterization at high magnification. High temperature optical microscope (EM201 HT163,
165 Hesse instruments, Germany) was used for optical dilatometry.

166

167 *2.5. Preparation of dye solution*

168 A synthetic dye (BTB), widely used in the textiles industry, was chosen as the targeted
 169 pollutant to gauge the sorption capacity of the fabricated specimens and their probable
 170 application in industrial wastewater purification. Stock solution was formed by dissolving
 171 exactly weighed amounts (1g) of dye in bi-distilled water to give a strength of 1000 mg·L⁻¹.
 172 From this solution, other solutions were prepared by successive dilutions when necessary.

173

174 *2.6. Batch adsorption experiments*

175 The adsorption experiment was performed by mixing 25 ml of dye solution and 0.65 g of
 176 adsorbent. The mixture was shaken at 200 rpm and placed in a centrifuge for 5 minutes at
 177 4000 rpm. The residual concentration of dye was determined by analysis of the supernatant at
 178 the wavelength that is consistent to the extreme absorbance, at 625 nm, using UV-visible
 179 spectrophotometer (Evolution 300, Thermo Scientific). Portion of adsorbed BTB was as per
 180 subsequent equation (1):

$$q_e(\text{mg}\cdot\text{g}^{-1}) = \frac{(C_i - C_e) \times V}{m} \quad (1)$$

181 where q_e is the quantity of dye adsorbed by bulk of adsorbent (mg·g⁻¹), C_i and C_e are
 182 respectively the initial and residual concentration of dye solution (mg·L⁻¹), m is the amount of
 183 adsorbent (g), and V is volume of solution (l).

184

185 The effect of various factors on the adsorption capacity were examined:

- 186 – The outcome of pH on the adsorption capacity, was evaluated by using various pH
 187 solutions ranging from 2 to 11. The solutions pH were adjusted by using diluted mix
 188 of HNO₃ and NaOH (0.1 N).
- 189 – Impact of contact time was studied from 5 to 300 min at three altered initial
 190 concentrations 50, 100 and 200 mg·L⁻¹. The obtained kinetic results were modelled by
 191 Elovich, Pseudo 1st order, Pseudo 2nd order and intraparticle diffusion kinetic models.
- 192 – The effect of initial concentration on dye adsorption capacity was investigated at
 193 20 °C with different dye mixes ranging from 10 to 1000 mg·L⁻¹. Freundlich, Langmuir
 194 and Temkin adsorption isotherm were utilized to represent the obtained data from
 195 adsorption isotherm study.

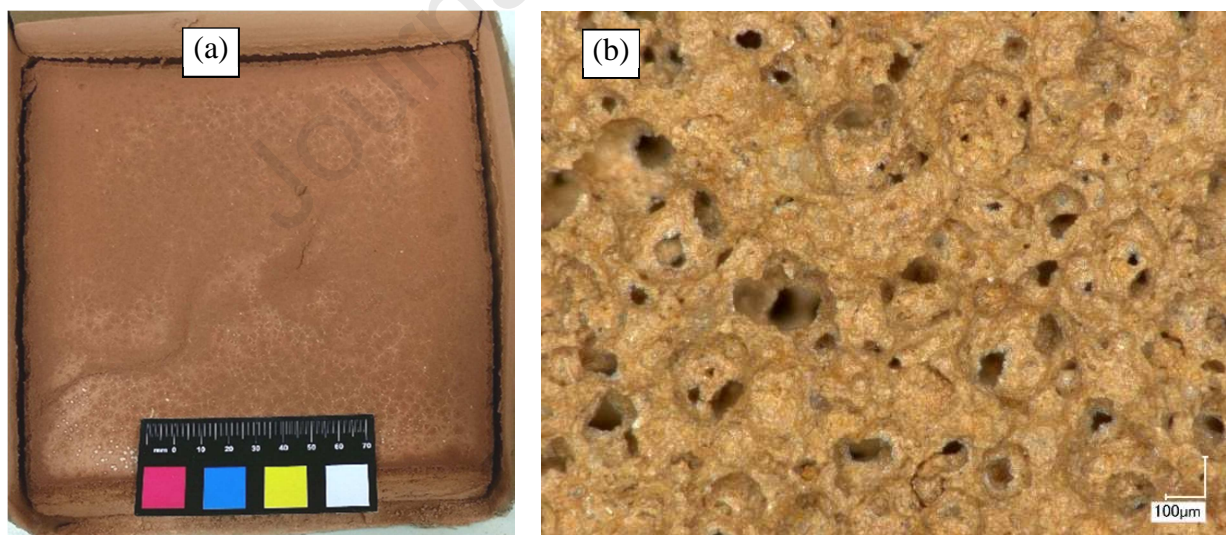
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197 **3. Results and discussion**

198 Results on clay foam are presented below for their physical, mechanical properties, and
199 adsorption performance.

200 3.1. Samples and pore morphology

201 The sample of the CF (MC-7 wt.%), shown in Fig. 1a, had no visible fractures or cracks
202 compared to the CF without MC addition (provided in supplementary information). The
203 microstructure demonstrates (Fig. 2b) an even pore distribution in the sample volume and the
204 presence of interconnected pores which points to an open cell material classification. The
205 sintering process slightly affected the pore size (due to shrinkage), but not the foam
206 morphology where a spherical-like and interconnected open cell microformation was noted
207 for all 12 samples series. The average cell diameter for the sintered CF was in the range of 50-
208 200 μm and the cell window measurement was 30-70 μm (Fig. 1b and 2a). The pore
209 interconnection was formed due to the thin suspension films between the bubbles rupturing
210 during dehydration since the dislocation of powder from the boundary is thermodynamically
211 inimical. The CF walls have a porous structure (Fig. 2b) due to the natural residual clay
212 porosity caused by water evaporation and by formation of lamellar clay structure. The porous
213 walls facilitate the liquid permeability through the structure. The MC addition did not play
214 significant role on the foam morphology and average cell sizes, which were in the 50-200 μm
215 range for all samples (5, 7 and 10 wt.% of MC addition).

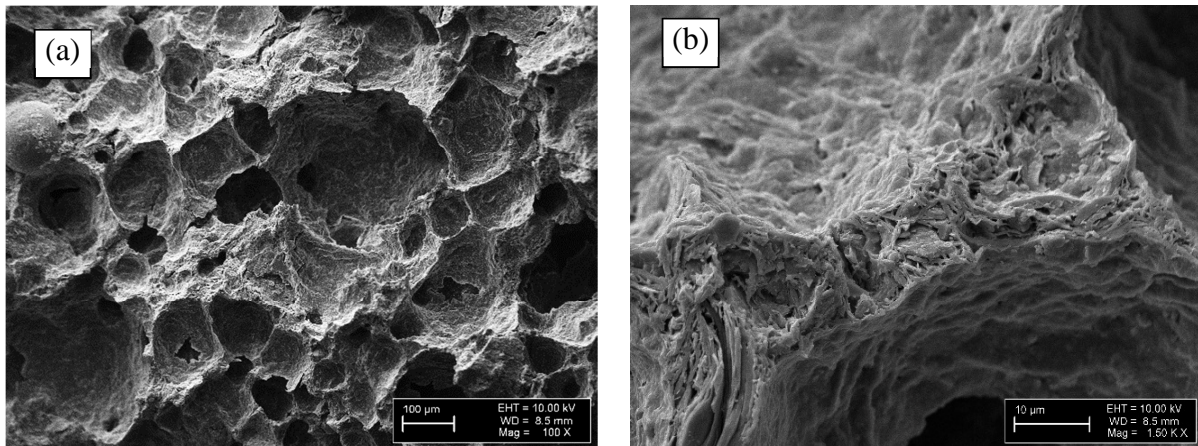


216
217

218 Fig. 1. CF with 7 wt.% MC content. a) green sample after drying; b) fired at 900°C, optical
219 microscope at magnification X200 times.

220

221 According to micro optical observations, with an escalation in the firing temperature the
 222 pore sizes decrease by 7-10% for the series sintered at 1050°C in comparison to 900°C. This
 223 could be explained by the natural clay shrinkage and densification during the firing.



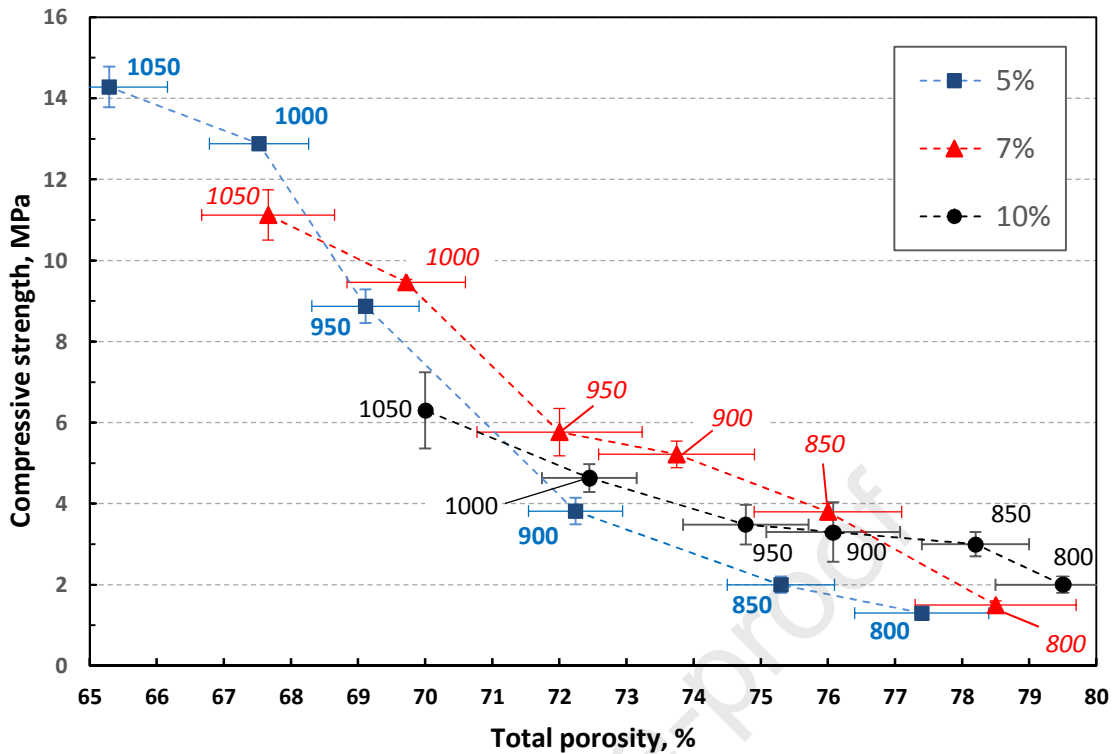
225 Fig. 2. SEM micrographs of the CF with 7 wt.% MC content, fired at 900°C
 226

227
 228

3.2. Physical properties

229 The compressive strength and total porosity of CF samples with diverse MC ratio are
 230 presented in Fig. 3. It can be perceived that the porosity augmented by 2-3% with an MC
 231 increase from 5 to 7 wt.% (for all sintering temperatures). At the same time, the porosity
 232 decreases by 2-2.5% with a firing temperature increase by 50°C for each series. The porosity
 233 dependence on the firing temperature exhibited a linear tendency. However, for the firing
 234 temperature increase from 950 to 1050 °C, the bulk density showed a steady increase for all
 235 compositions, from 0.80 to 0.90 g·cm⁻³, from 0.73 to 0.84 g·cm⁻³, and from 0.66 to 0.75 g·cm⁻³
 236 for 5, 7 and 10 wt.% MC content, respectively. For porous ceramics the bulk density has a
 237 direct correlation with shrinkage, the higher the sintering temperature the denser the ceramic
 238 particles pack and at the same time pore merging can take place.

239 Compressive strength increases for all series, with the firing temperature increase.
 240 However, the intensity of increase is highly dependent on the MC content. At 900°C clay
 241 particles could be bonded and in this case, the amount of MC plays a more significant role.
 242 The result of 7 wt.% MC composition shows a higher value of compression strength at 900°C.
 243 Overall, the low mechanical properties of 10 wt.% MC sample could be explained by the
 244 lower mechanical properties of the glass phase. The total porosity varies almost linearly with
 245 sintering temperature increase for all compositions (Fig. 3).
 246



247 Fig. 3. Dependence of the compressive strength on the firing temperature of samples with 5,7
 248 and 10 wt.% of MC loading at different firing temperatures. MC ratio is indicated in the
 249 legend. The firing temperature is indicated in °C.
 250

251 In view of definite mechanical properties - specific strength (Φ/Δ) is of vital significance in
 252 lightweight product design and determination of the best material (Ashby, 2011). From the
 253 engineering point of view, lightweight panels could be described and evaluated by specific
 254 strength Φ/Δ ($\text{MPa}\cdot\text{g}^{-1}\cdot\text{cm}^{-3}$) (Novais et al., 2015; Ponsot et al., 2015). However, from an
 255 energy efficiency viewpoint it is necessary to consider one more aspect – the temperature and
 256 time required for the material production. In the case of investigated natural clay foam with
 257 the addition of 5 wt.%, 7 wt.% and 10 wt.% of ground MC, optimal parameters were found
 258 for 7 wt.% MC. This composition has the highest compressive strength in the 900°C series -
 259 5.2 MPa. The composition with 5 wt.% MC, sintered at 1000°C has the highest compressive
 260 strength in the 1000°C series – 12.9 MPa, and the second highest value of compressive
 261 strength (12.6% less than the highest value – 14.3 MPa) in all the series. Therefore
 262 composition with 5 wt.% MC, sintered at 1000°C, is better for the construction application
 263 (with higher mechanical properties). The record of absolute (MPa) and specific ($\text{MPa}\cdot\text{g}^{-1}\cdot\text{cm}^{-3}$)
 264 strength of the 5-10 wt.% MC loaded clay foam are provided in Table 1.
 265

266 Table1. The absolute Φ (MPa) and specific Φ/Δ (MPa·g⁻¹·cm⁻³) strengths of MC-clay
267 foam.

Firing temperature	MC content, wt.%					
	5 wt.%		7 wt.%		10 wt.%	
	Φ	Φ/Δ	Φ	Φ/Δ	Φ	Φ/Δ
800 °C	1.3±0.2	1.9	1.5±0.1	2.3	2.0±0.2	3.6
850 °C	2.0±0.2	2.9	3.8±0.2	5.8	3.0±0.3	5.2
900 °C	3.8±0.3	5.3	5.2±0.3	7.7	3.3±0.7	5.3
950 °C	8.9±0.4	11.1	5.8±0.6	7.9	3.5±0.5	5.3
1000 °C	12.9±0.1	15.3	9.5±0.1	12.0	4.6±0.3	6.5
1050 °C	14.3±0.5	15.8	11.1±0.6	13.2	6.3±0.9	8.4

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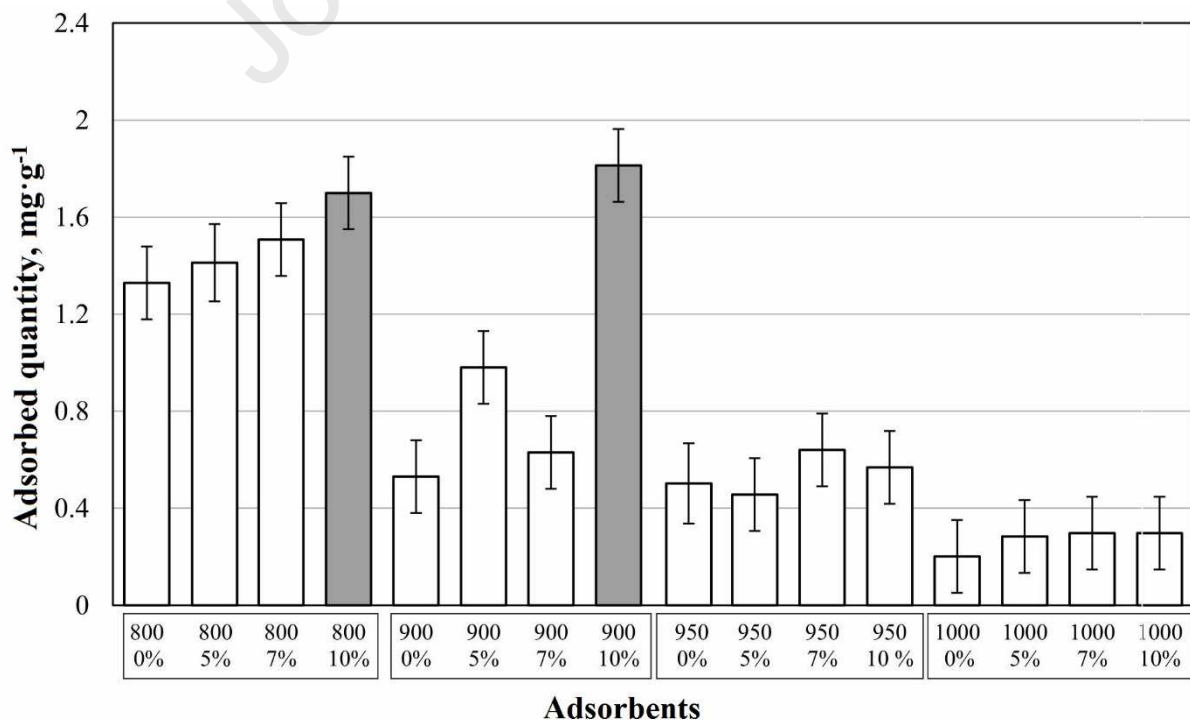
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270 3.3. Effect of firing and MC content on the adsorption capacity

271 Preliminary adsorption tests were conducted to evaluate the outcome of firing
272 temperatures (800, 900, 950, and 1000 °C) and glass contents (5, 7 and 10 wt.%).

273 As demonstrated in Fig. 4, the highest adsorption capacity was observed for samples prepared
274 at relatively low temperatures (800, 900°C) containing 10 wt.% of glass content. The
275 uttermost adsorption capacity registered was 1.7 and 1.8 mg·g⁻¹ respectively for MC10-800
276 and MC10-900. The lowest adsorption capacity was observed at high firing temperatures.
277 Therefore, compositions MC10-800 and MC10-900 were selected as adsorbents for further
278 evaluation of the adsorption process of BTB dye.

279



280

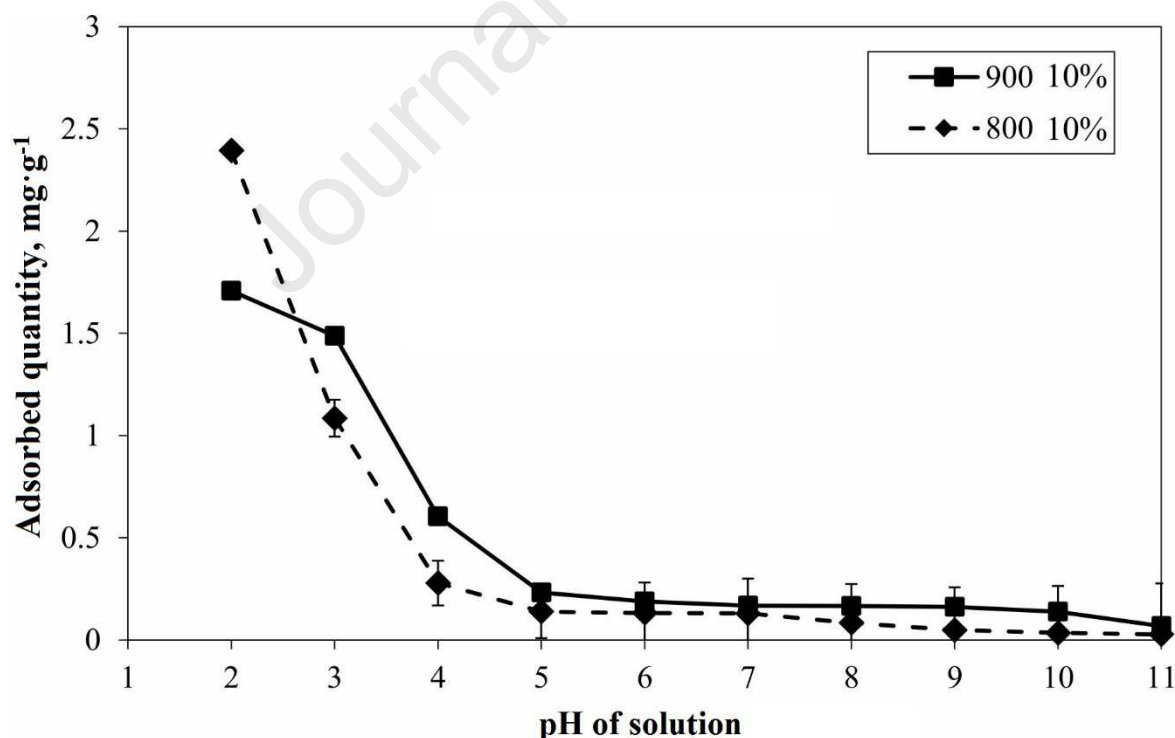
281 Fig. 4. Preliminary adsorption test of different prepared materials.

282 3.4. Influence of pH

283 The pH of mix significantly influences the adsorption capacity of dye onto MC10-800 and
 284 MC10-900 as shown in Fig. 5. Indeed, the adsorption capacity augmented when the pH of
 285 solution reduced, with the maximum adsorption capacity for both materials being observed at
 286 pH 2. Adsorption capacity of 2.4 and 1.7 mg·g⁻¹ was obtained, respectively, for MC10-800
 287 and MC10-900. These capacities decreased when the pH of solution increased, achieving the
 288 lowest values at pH 5.

289 As explained by earlier study (Aguedal et al., 2018), the high amount of BTB dye
 290 adsorbed onto raw and thermally treated diatomite, in the acidic range, is due to the
 291 protonation influence of superficial functional groups of the adsorbents. This effect generates
 292 a high electrostatic attraction among the positively charged adsorbent surface and the dye that
 293 seems anionic character. However, in the alkaline pH range, hydroxyl ions compete with the
 294 molecules of dye to occupy the same available active adsorption sites, which translates into
 295 the lowest amount of dye being adsorbed in this range. Hence, a pH 2 is used in the kinetic
 296 and isotherm adsorption studies.

297



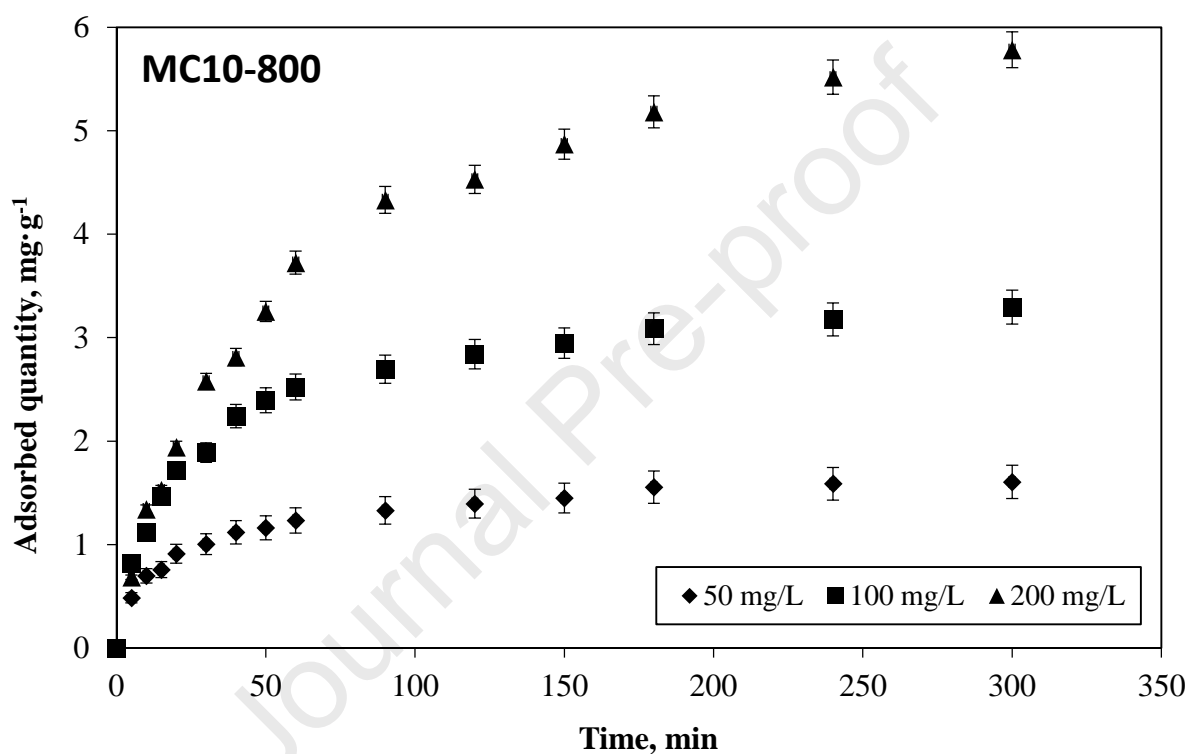
298 Fig. 5. pH effects on the adsorption capacity of dye at MC10-800 and MC10-900°C.
 299

300

301 3.5. Influence of contact time

302 The rate at which the adsorbent is able to uptake the ascorbate is one of the most
303 significant parameters that governs the appropriateness for the purpose of water quality
304 control. The kinetic study was done to analyse the mechanism and determine the efficiency of
305 the adsorption process. Three concentrations of dye (50, 100 and 200 mg·L⁻¹) were utilized to
306 analyse the adsorption kinetic of BTB dye onto MC10-800 and MC10-900 at different time
307 intervals, between 5 and 300 min, and at an optimised pH. The results are depicted in Fig. 6
308 and Fig. 7.

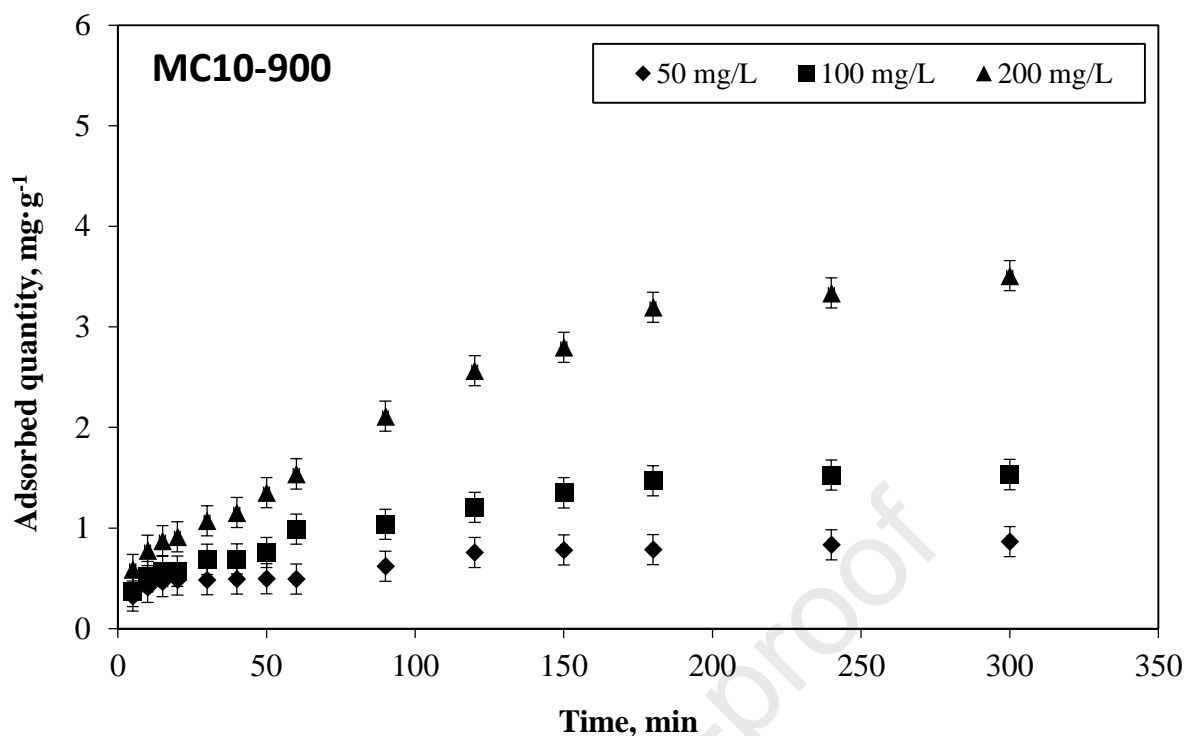
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310

311

Fig. 6. Sorption kinetics for MC10-800.



312

313

Fig. 7. Sorption kinetics for MC10-900.

314 It was observed, for all selected dye concentrations, that the adsorption capacity of BTB
 315 dye augmented with, surge in contact time and then the adsorption rate reduced and tends
 316 towards equilibrium. In addition, the surge in the initial dye concentration increased the
 317 exclusion efficiency of dye and significantly increased the equilibrium time. At lower
 318 concentrations, the number of available adsorption sites is higher than the number of dye
 319 molecules in solution, which lead to equilibrium rapidly. However, at higher concentrations,
 320 the amount of dye molecules experiences high gradient of concentration at the liquid-solid
 321 interface which increase the deep molecules diffusion to the inner part of MC10-800 and
 322 MC10-900 adsorbents, thus explaining the rise in adsorption capacity of dye and equilibrium
 323 time (El Haddad et al., 2014). The same observations have been reported in other work (Iddou
 324 et al., 2011; Novais et al., 2018).

325 Adsorption kinetic behaviour of BTB dye onto MC10-800 and MC10-900 was studied
 326 using the experimental kinetic specifics and analysed by the Elovitch, pseudo-first order,
 327 pseudo-second order and intraparticle diffusion kinetic models (see Table 2). The results of
 328 the correlation coefficient and the calculated constant bounds for each model are summarised
 329 in Table 3.

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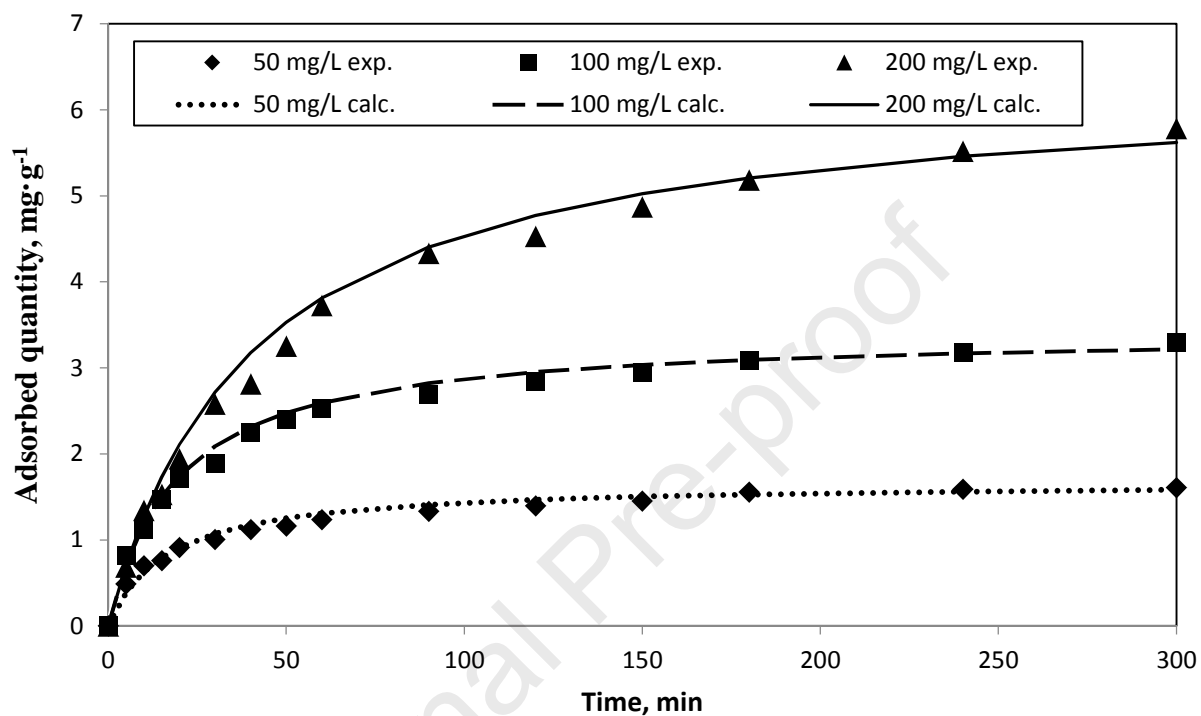
Table 2. List of non-linear equations of the used kinetic models and their linearized expressions.

Kinetic models	Equations	Linear expression	Plot	References
Elovich	$q_t = \beta_E \times \ln(\alpha_E \times \beta_E \times t)$	$q_t = \beta \times \ln(\alpha \times \beta) + \beta \times \ln(t)$	q_t vs. $\ln t$	(Zhang and Stanforth, 2005)
Pseudo 1 st order	$q_t = q_e [1 - \exp(-k_{1p} \times t)]$	$\ln(q_e - q_t) = \ln(q_e) - k_{1p} \times t$	$\ln(q_e - q_t)$ vs. t	(Aravindhana et al., 2007)
Pseudo 2 nd order	$q_t = k_{2p} \times Q_e^2 \times t / (1 + k_{2p} \times Q_e \times t)$	$t/q_t = (1/k_{2p} \times Q_e^2) + (t/Q_e)$	t/q_t vs. t	(Ho and McKay, 1999)
Intra-particle diffusion	$q_t = k_p \times t^{0.5}$	$q_t = k_p \times t^{0.5}$	q_t vs. $t^{0.5}$	(Arami et al., 2008)

q_t and q_e are respectively the adsorbed quantity of dye at time t , and at equilibrium ($\text{mg} \cdot \text{g}^{-1}$). k_{1p} , k_{2p} and k_p are respectively the kinetic model constant of pseudo-first order (min^{-1}), pseudo-second order ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$) and intraparticle diffusion ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-0.5}$); α ($\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{-1}$) and β ($\text{g} \cdot \text{mg}^{-1}$) are the Elovich kinetic model constants.

1 From the high values of correlation coefficient R^2 , the adsorption kinetics of BTB dye are
2 well matched by the pseudo-second order kinetic model for all adsorbents. In addition, the
3 calculated adsorption capacities by this model are similar to those obtained in batch mode test
4 (see Fig. 8 and Fig. 9).

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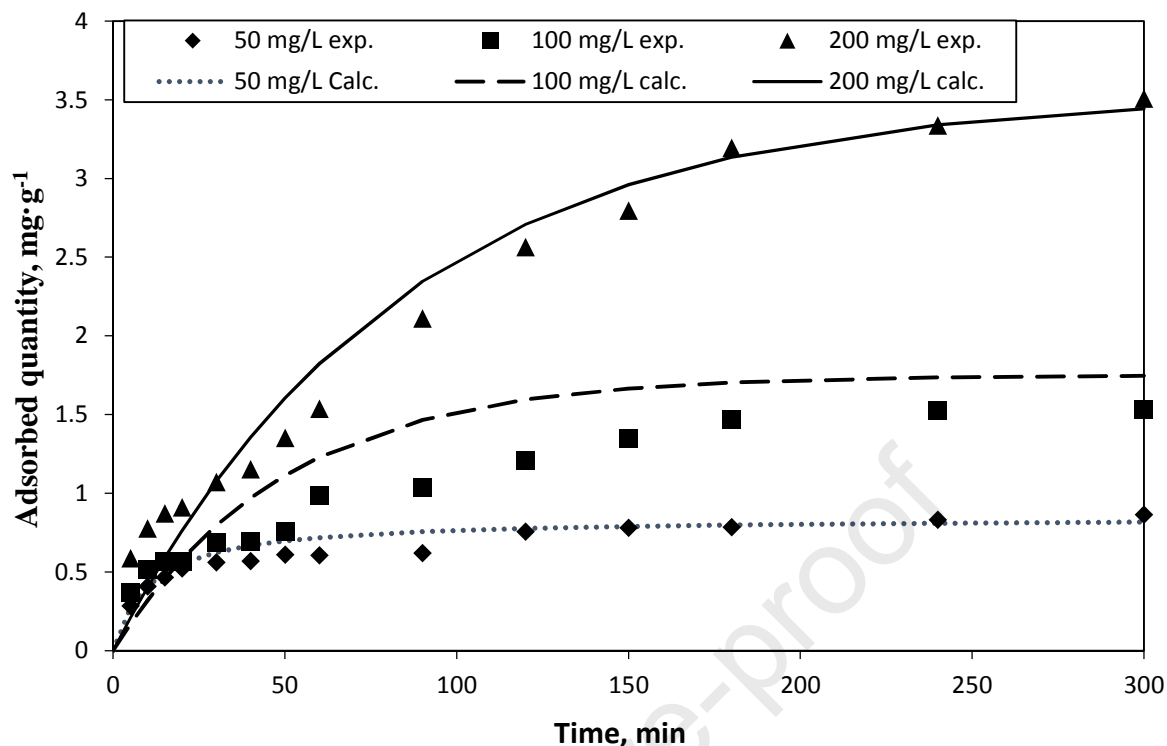
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8 Fig. 8. The adsorption amount of dye onto MC10-800 obtained experimentally and fitted
9 pseudo-second order kinetic model.

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15 Fig. 9. The adsorption amount of dye onto MC10-900 obtained experimentally and fitted
 16 pseudo-second order kinetic model.

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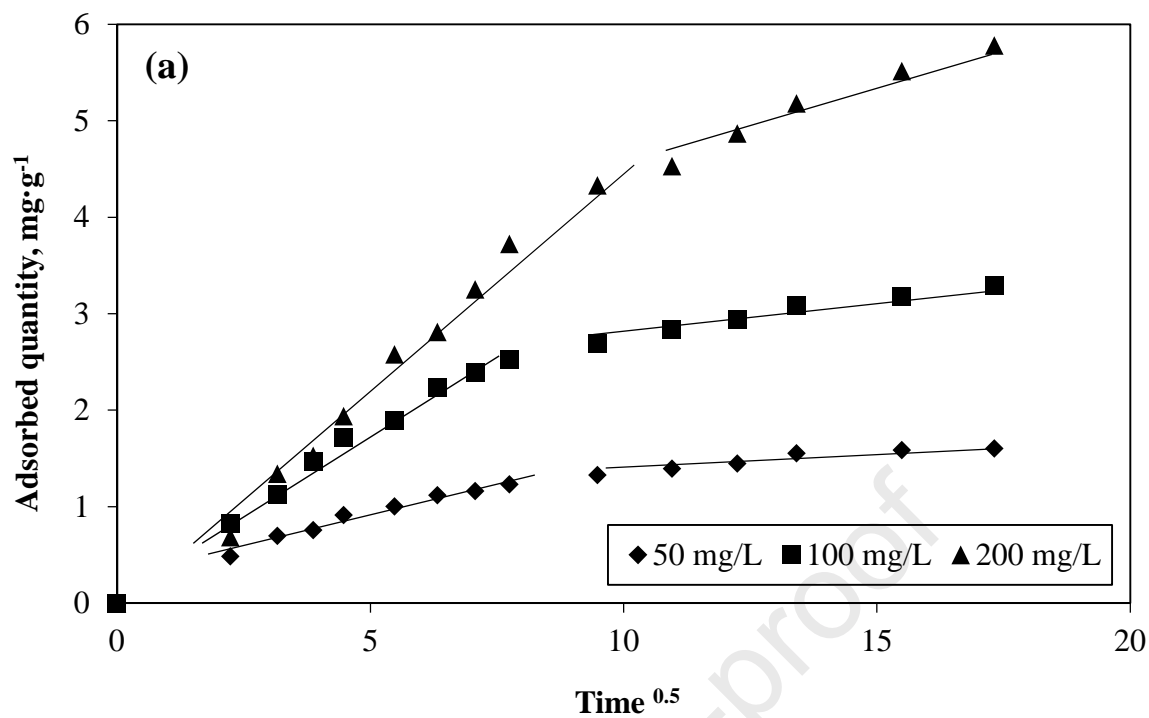
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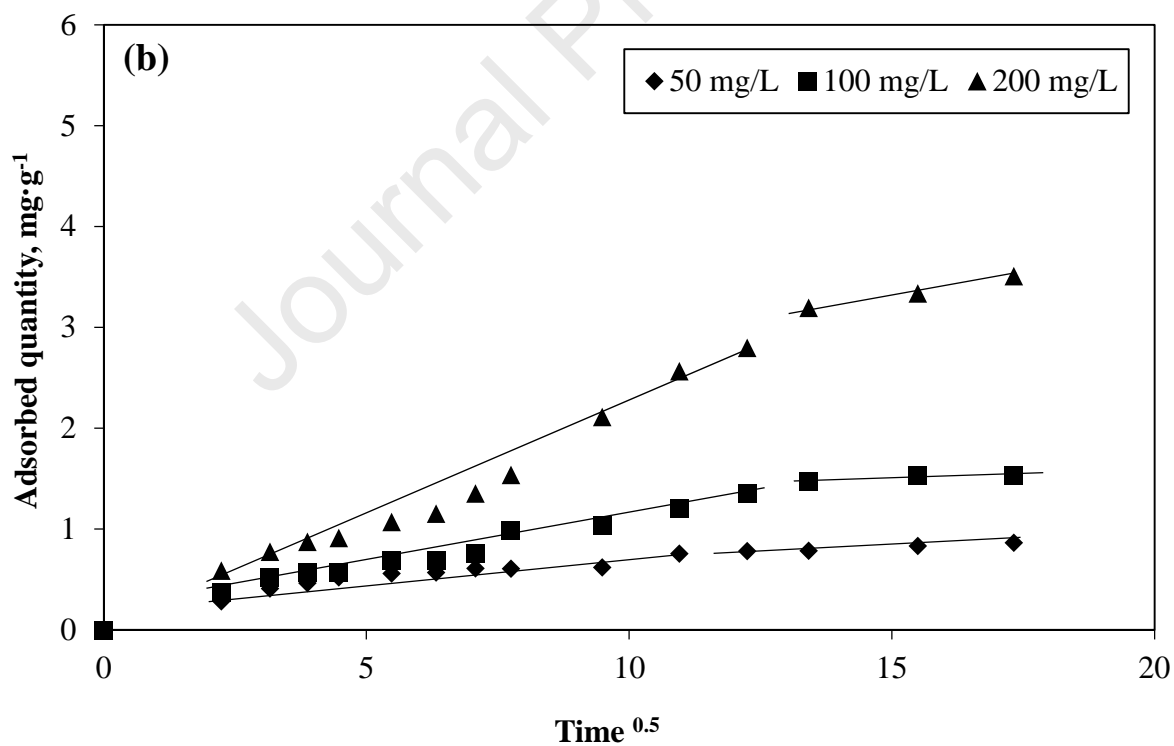
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Matching of experimental results to the pseudo-second order kinetic model indicates that the chemisorption could be the rate-controlling step on the first stage as well as at low concentrations. However, the assumption of molecular diffusion could be one more mechanism involved in the removal process of BTB dye onto MC10-800 and MC10-900, which is justified using intraparticle diffusion kinetic model. Indeed, as reported by Weber and Morris (W. Weber, J. Morris, 1963), if intraparticle diffusion is the dominant step in the adsorption method of BTB dye onto MC10-800 and MC10-900, then a plot of q_t versus $t^{0.5}$ ought to be linear and advance along the origin (Behnamfard and Salarirad, 2009). From Fig. 10a and Fig. 10b it can be seen that the plot is often multilinear (two linear steps) signifying that the intraparticle diffusion is by no means the rate-limiting step for the entire adsorption process. The initial linear step confirms the fast adsorption during the first time of contact. As the reaction constant for the intraparticle diffusion k_p increase with the initial dye concentration, the slowdown in the second stage is certainly owing to the resistance diffusion of dye molecules into the interior adsorption available sites (Bhatti and Nausheen, 2014).



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Fig. 10 Intraparticle diffusion kinetic model plots of BTB dye adsorption at different initial concentration onto (a) MC10-800, (b) MC10-900.

Table 3. Kinetic model parameters for MC10-800 and MC10-900 samples.

Kinetics model	Parameters	MC10-800			MC10-900		
		50 mg·L ⁻¹	100 mg·L ⁻¹	200 mg·L ⁻¹	50 mg·L ⁻¹	100 mg·L ⁻¹	200 mg·L ⁻¹
Elovich	β	0.288	0.61	1.136	0.14	0.28	0.66
	α	3.76	1.94	0.37	9.67	1.97	0.52
	R^2	0.996	0.991	0.953	0.98	0.93	0.86
Pseudo-first order	q_e	1.28	2.11	4.961	0.53	1.75	3.53
	k_{1p}	0.017	0.013	0.012	0.012	0.02	0.012
	R^2	0.945	0.928	0.96	0.94	0.89	0.98
Pseudo-second order	q_e	1.67	3.42	6.38	0.88	1.68	4.1
	k_{2p}	0.036	0.015	0.004	0.067	0.017	0.003
	R^2	0.995	0.995	0.983	0.99	0.97	0.91
Interparticle diffusion	k_p	0.038	0.177	0.352	0.041	0.09	0.22
	R^2	0.861	0.871	0.954	0.85	0.96	0.98

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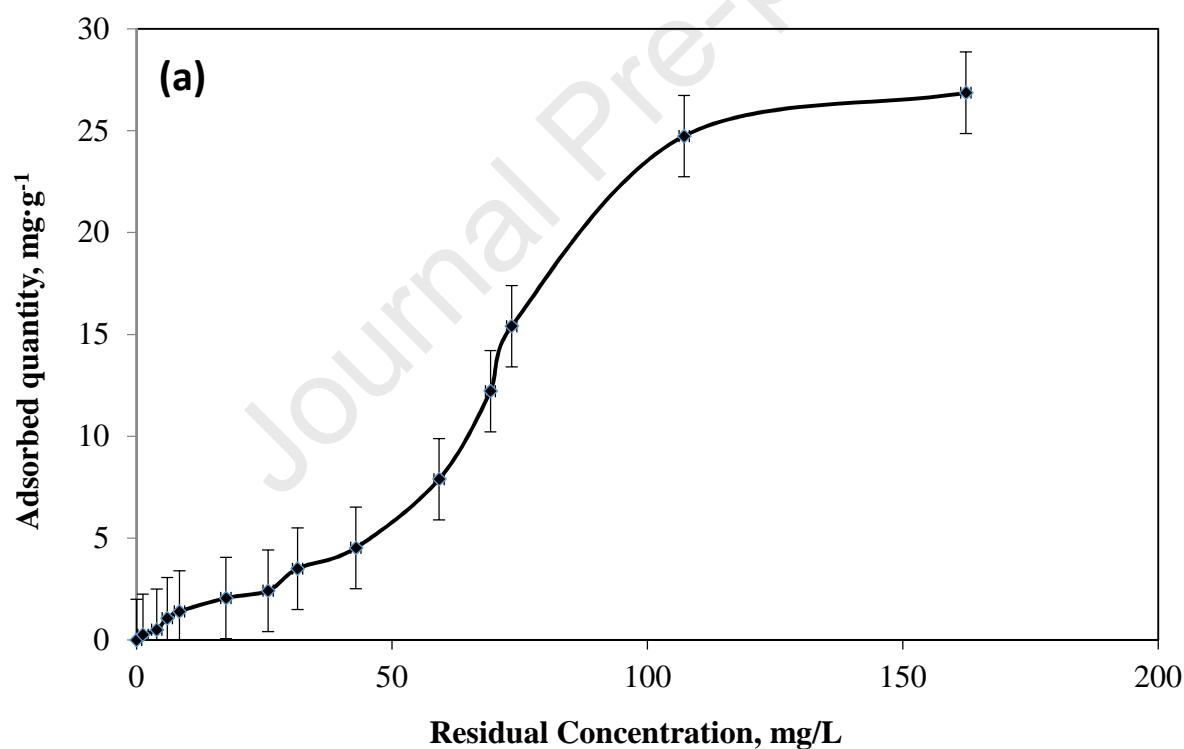
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44 3.6. Adsorption isotherm

45 The adsorption equilibrium isotherm was assessed by studying the outcome of initial
46 dye concentration at room temperature and optimized value of pH and contact time (pH =
47 2, equilibrium time of 2h). Tests were performed at varying dye concentrations from 10 to
48 900 mg·L⁻¹. Fig. 11a and Fig.11b depict, that the rise in the early dye concentration causes
49 a rapid growth in the amount of dye adsorbed onto MC10-800 and MC10-900, and
50 gradually increases until the equilibrium and/or saturation state of MC10-800 and MC10-
51 900 adsorbent is reached. The same phenomenon was observed by Kousha et al (2012).
52 As reported by Novais et al (2018), that the mass transfer resistances at the solid-liquid
53 interface is overcome by increasing the preliminary dye concentration, which provide
54 significant driving force enhancing the adsorption process. Hence, a higher initial
55 concentration could lead to adsorbent saturation.

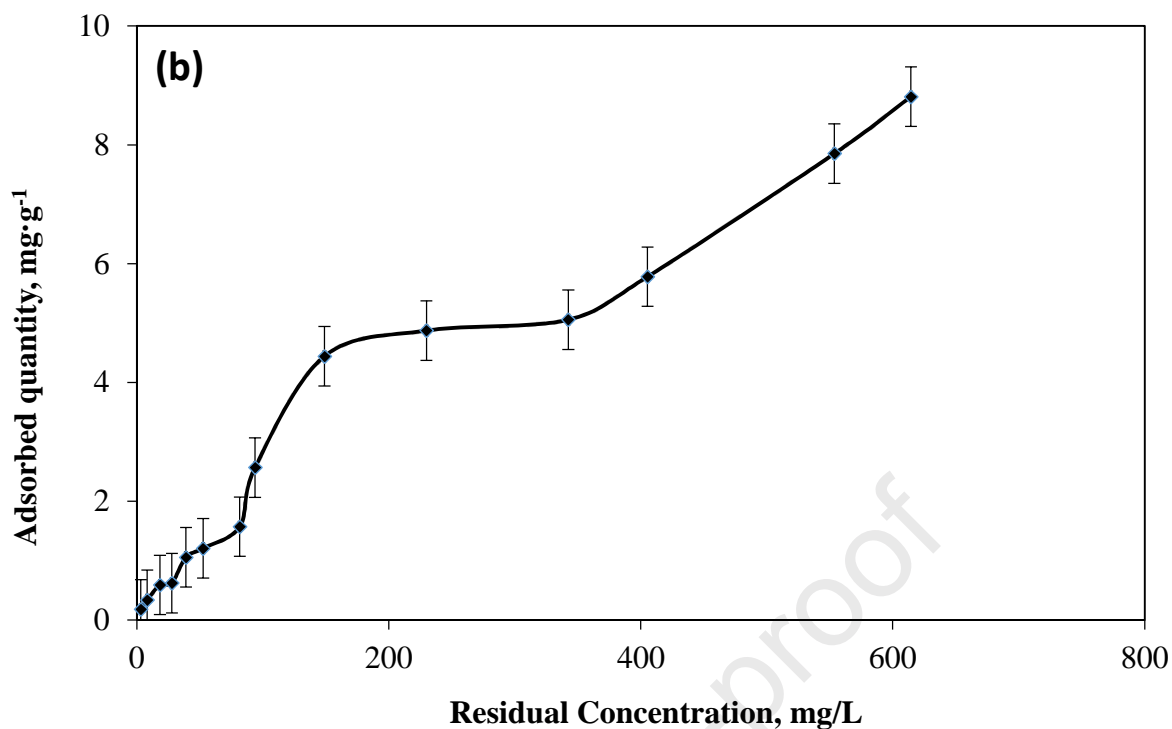
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60 Fig. 11. Effect of initial dye concentration (a) MC10-800 and (b) MC10-900.

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63 Equilibrium isotherm equations characterize the adsorption mechanism of BTB dye
64 onto MC10-800 and MC10-900 adsorbents. Observed data were fitted by Langmuir,
65 Freundlich and Temkin isotherm model (see Table 4). In order to decide if the isotherm model
66 is suitable to describe the experimental results, high correlation coefficients (R^2) for the best
67 fitting isotherm model can be referred. Table 5 summarises all the correlation coefficient R^2
68 values obtained from each of the models applied in the adsorption study of BTB dye onto
69 MC10-800 and MC10-900.

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Table 4: List of non-linear equations of the used isotherm models and their linearized expressions.

Isotherm model	Equations	Linear expression	Plot	References
Freundlich	$q_e = K_F \times (C_e)^{1/n}$	$\ln(q_e) = \ln(K_F) + (\ln(C_e)/n)$	$\ln(q_e)$ vs. $\ln(C_e)$	(Chan et al., 2012)
Langmuir	$q_e = (K_L \times Q_m \times C_e) / (1 + K_L \times C_e)$	$1/q_e = (1/K_L \times q_m \times C_e) + (1/q_m)$	$1/q_e$ vs. $1/C_e$	(Secula et al., 2011)
Temkin	$q_e = q_m \times \ln(K_T \times C_e)$	$q_e = q_m \times \ln(K_T) + q_m \times \ln(C_e)$	q_e vs. $\ln(C_e)$	(Behnamfard and Salarirad, 2009)

q_e ($\text{mg} \cdot \text{g}^{-1}$) and C_e ($\text{mg} \cdot \text{L}^{-1}$) are respectively the equilibrium adsorbed quantity and equilibrium concentration, K_F ($\text{mg} \cdot \text{g}^{-1}$) Freundlich isotherm constant ($\text{mg} \cdot \text{g}^{-1}$), $1/n$ Freundlich exponent, q_m ($\text{mg} \cdot \text{g}^{-1}$) maximal adsorption quantity, K_L ($\text{L} \cdot \text{mg}^{-1}$) Langmuir isotherm constant, K_T ($\text{L} \cdot \text{mg}^{-1}$) Temkin isotherm constant.

Table 5. The isotherm model parameters for MC10-800 and MC10-900.

Isotherm model	Parameters	MC10-800	MC10-900
Freundlich	K_F	0.28	0.19
	n	1.19	1.82
	R^2	0.87	0.76
Langmuir	K_L	0.023	0.018
	q_m	8.80	3.16
	R^2	0.97	0.96
Temkin	K_T	0.28	9.98
	q_m	4.36	1.32
	R^2	0.60	0.75

From Table 5, it may be perceived that the adsorption isotherm data of BTB dye onto MC10-800 and MC10-900 are best matched by the Langmuir isotherm model giving the highest R^2 comparing to Freundlich and Temkin models. Underpinning the Langmuir assumptions, the adsorption molecules of BTB dye onto MC10-800 and MC10-900 occurred in monolayer at a homogeneous adsorbent surface. Thus chemisorption is the main mechanism controlling the whole adsorption process (Caner et al., 2009).

The increasing sorption ability of the MC10-800 material can be explained by the high porosity of CF due to a lower firing temperature. This is supported by the high temperature microscopy results (Fig. 12a and 12b), where it is clearly visible that at temperature above 860°C, the glass changed from solid conditions, corresponding to low-viscosity state and transfers into a semi-liquid phase. At 900 °C glass has much lower viscosity. Beyond 900 °C glass addition promotes Liepa clay sintering, which decreases porosity and increases shrinkage which in good correlation with obtained strength-porosity results for studied porous ceramics in this research.

This is supported by the dilatometry measurements which demonstrated a rapid increase of the shrinkage (Fig. 12a) after 860°C. At the same time, at 860°C, the glass completely transferred to semi-liquid conditions (Fig. 12b) and started working as a ceramic particle binder. Thereby, conditions are optimum at 800°C for MC utilisation with maximum porosity and results in maximum adsorption in the specimen.

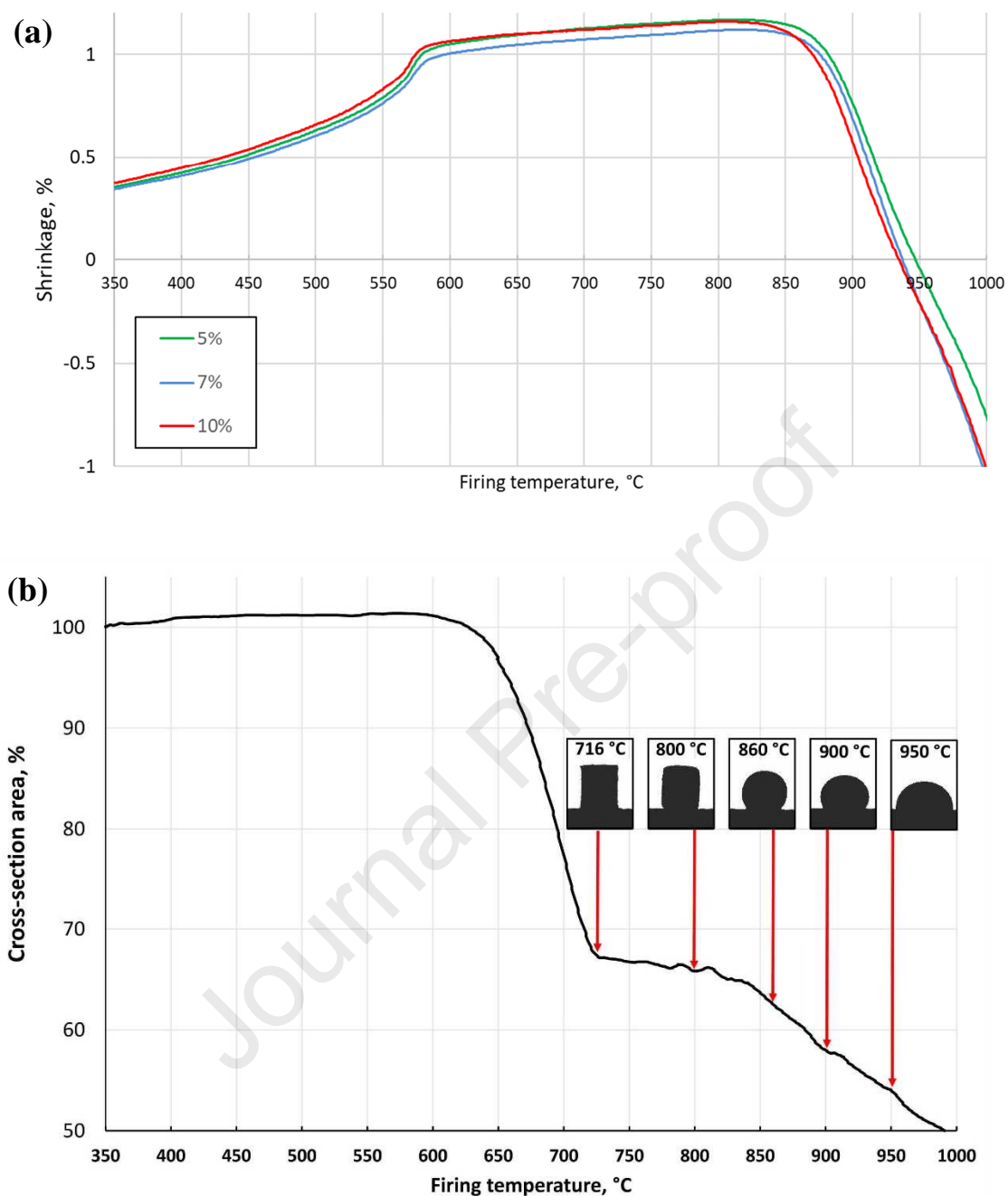


Fig. 12. (a) Dilatometry measurements of the MC5, MC7, MC10 (b) high-temperature microscopy measurements of the glass prism cross-section during firing.

3.7. Benchmarking of results and possible mechanism of dye adsorption

Removal of BTB with other adsorbents used by other researchers has been compared in Table 6. Values reported in the table implies that adsorption capacity achieved in this study is comparable with other studies in literature. However it may be noticed while others materials

(e.g. activated carbon) are carbon based and may be for single use whereas ceramic material obtained in this work can be reused for several cycles after thermal treatment or it can be used as construction material (Petrella et al., 2018). Thus, it is cost efficient and environment friendly.

Table 6. Removal of the BTB dye with different adsorbents as reported in literature.

Adsorbent	Adsorption capacity (mg.g ⁻¹)	Reference
MC10-800	26.8	Present study
Diatomite ^a	27.8	(Aguedal et al., 2018)
Activated sludge	38.2	(Djafer et al., 2016)
Chitosan/zeolite A	305.8	(Nešić et al., 2013)
Brown macroalga	35.6	(Kousha et al., 2012)
Pine Cone	37.4	(Mahmoodi et al., 2011)
Humins immobilized on silica	19.4	(Jesus et al., 2011)
Activated carbons ^b	66.8	(Ahmad and Rahman, 2011)
Anaerobic sludge	117.1	(Caner et al., 2009)
	100.0	(Kargi and Ozmihiç, 2005)
Activated carbons ^c	3.4	(Amin, 2008)
Chitosan-crosslinked beads	30.0	(Kimura et al., 2002)

^a : Treated at 600°C

^b : Prepared from coffee husk

^c : Prepared from bagasse pith

Based on literature and according to the effect of pH on the adsorption process, possible scheme of the BTB dye adsorption mechanism on the fired clay-glass surface is as follows. All the reactive dyes behaves similarly in aqueous media (Awasthi et al., 2020). As noticed from molecular structure of BTB dye (Fig. 13), it will be ionized in aqueous solution, by the presence of sulfonate group (SO³⁻) groups and the rupture of the OH bond leading to an overall negative charged aqueous solution. On the other hand, in acidic range, the protonation effect conduct to charge positively all the functional groups on the surface of ceramic clay (Al-Ghouti et al., 2009). Hence, a strong electrostatic attraction is created between the dye molecules and the surface of ceramic clay according to the scheme (Fig. 14). Once, bonds structure is occupied, further links could occur between the dye molecules via intermolecular attraction such as Van der Waals and hydrogen bonds (Ahmed and Jhung, 2017). Additionally, the O–H or oxygen bonds present on CF (Yang et al., 2020) may probably form an n– π bonding with the aromatic ring of BTB dye (Olusegun and Mohallem, 2020). The decrease of the adsorption capacity at high pH is due to, at alkaline range, the hydroxyl ions will be adsorbed strongly on the active site onto ceramic clay competing thereby with the

BTB dye molecules, hence leading to the high repulsive interactions for the BTB dye molecules (Awasthi et al., 2020).

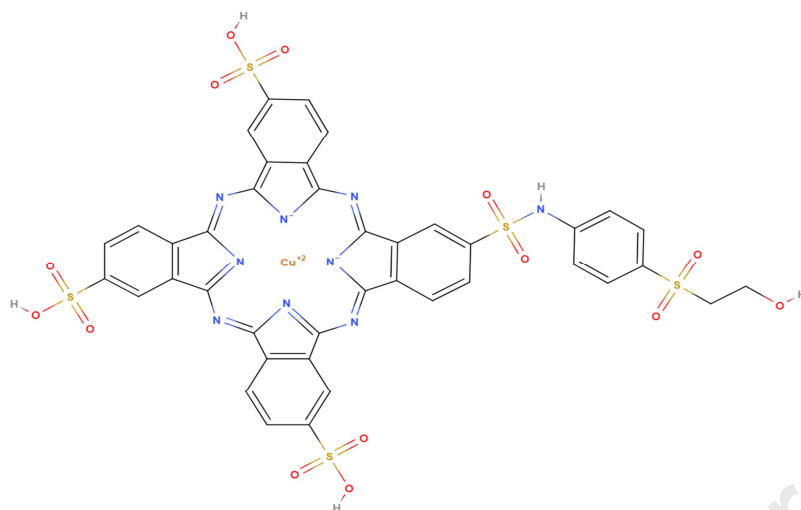


Fig. 13. BTB dye chemical structure (ChemicalBook, 2020).

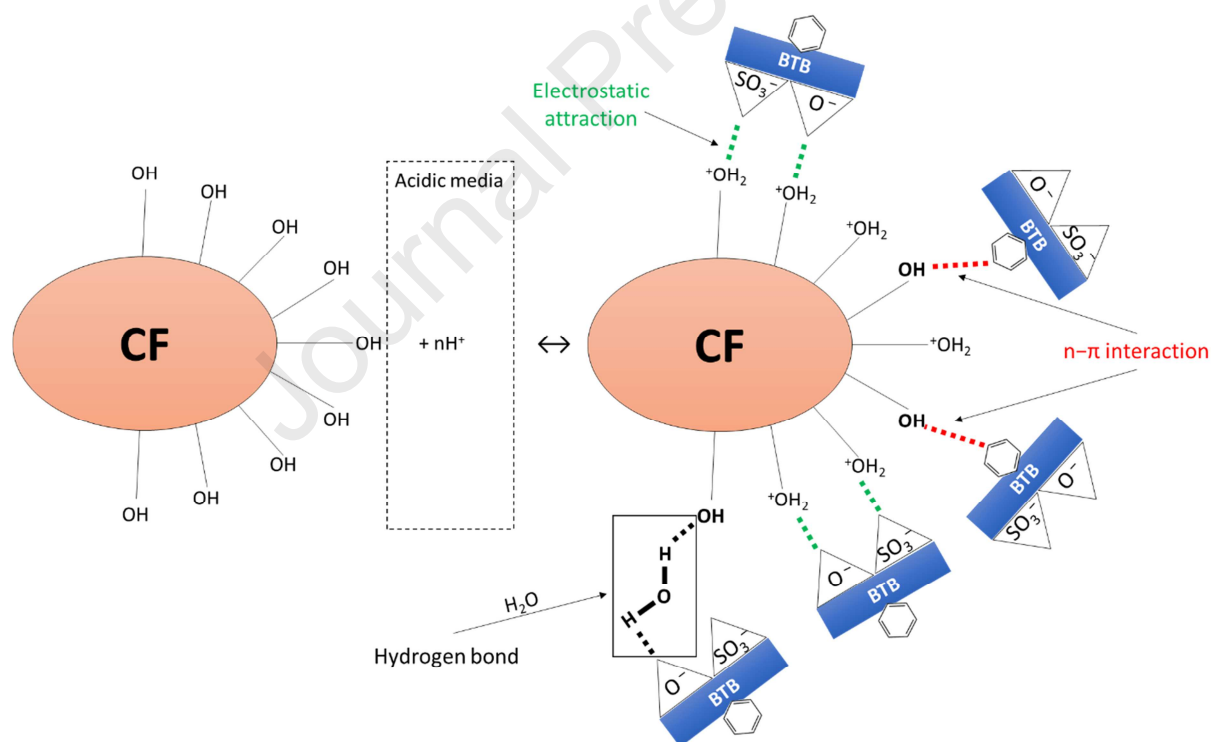


Fig. 14. Scheme of the possible adsorption process mechanism.

Conclusion

This novel multidisciplinary research paper reports experimental aspects involved in the development of new material (potentially useful as a component of industrial water filtration system). The material fabricated is highly porous clay ceramic foam containing cullet and is

obtained by direct foaming and cavitation disperser. Due to the relatively simple technology of ceramic clay foam production, with careful technological development, the technological uptake of the method is sustainable and it addresses the pressing international need of bringing environmental-friendliness in industrial production. Future development of the clay-glass ceramics should be directed towards lowering of firing temperature below 800 °C and improved glass milling efficiency to obtain finer particles with higher specific surface area to achieve greater sorption ability.

During this investigation, it was discovered that the mechanical properties improve with the increasing glass content and perform best when the fired temperature is in the range of 800-900°C. Other broad conclusions drawn were as follows:

- Benchmarking done against the published literature showed the newly proposed material possess a desirable adsorption capacity of 26.8 mg.g⁻¹ comparable to other research materials developed and reported to date. Furthermore, the open cell ceramic foam promotes a cleaner alternative.
- Experimental results indicated that the glass transferred to liquid phase above 860°C and started promoting intensive bonding with ceramic particles. This bonding contributes towards higher compressive strength at higher sintering temperatures.
- A pH value of 2 was found most optimal to attain the maximum adsorption capacities of up to 13%. The equilibrium time was affected by the intensification in the initial dye concentration.
- The sorption kinetic process followed the pseudo-second order kinetic model, indicating potential chemical relations between the sorbent and the sorbate. The Langmuir isotherm model described the experimental results well compared to other empirical models. Thus, chemisorption was discovered to be the driving mechanism controlling the adsorption process.

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Highlights

- Direct foaming method for formulating an open cell ceramic material.
- Maximum sorption capacity of $26.8 \text{ mg} \cdot \text{g}^{-1}$ for sample fired at $800 \text{ }^\circ\text{C}$.
- Cullet transferred from glassy to liquid conditions and promoted intensive bonding.
- Chemisorption is the main mechanism controlling the whole adsorption process.

Journal Pre-proof

Andrei Shishkin: Conceptualization, Methodology, Experimentation, Writing - original draft, Writing - review & editing. **Hakim**

Aguedal: Conceptualization, Methodology, Formal analysis, Writing - original draft, Writing - review & editing. **Gaurav**

Goel: Conceptualization, Writing - original draft, Writing - review & editing. **Julite Peculevica:** Methodology, Experimentation,

Investigation. **Darryl Newport:** Methodology, Writing - review & editing.

Jurijs Ozolins: Project administration, Data curation.

Journal Pre-proof

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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