

1 **Effect of phase modifiers on boron removal by solvent extraction using 1,3 diolic**
2 **compounds**

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19

20 **ABSTRACT**

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23 **BACKGROUND:** Among the polyols, 1,3 diols, are selective and efficient extractants
24 for boron but, significant solubility of these into aqueous solutions restricts their
25 application in large scale separation processes. Solvent modifiers can be used to reduce

26 extractant loss to the aqueous phase. Efficiency of the diolic extractants on boron
27 separation have been tested in presence of different solvent modifiers.

28 **RESULTS:**

29 Compared to other modifiers studied, decanol gives better stability to diolic molecules
30 in the Kerosene phase and exhibit lesser negative effect on the extraction of boron. Next
31 to decanol, the low viscous 2,6-dimethyl-4-heptanone can be used as solvent modifier.

32 In the successive extraction studies, 2-butyl-2-ethyl-1,3-propanediol (BEPD) showed
33 better stability among the diols studied in presence of decanol in the organic phase

34 **CONCLUSION:**

35 Quantitative removal of boron could be achieved from industrial waste solutions such as
36 a spent phosphate passivation bath and a cutting fluid, using 0.6 M BEPD and 0.6 M
37 2,2,4-trimethyl-1,3-pentanediol (TMPD) in 25% decanol/Kerosene organic phase.

38

39 **Keywords:** boron removal; liquid-liquid extraction; diolic compounds; solvent
40 modifiers.

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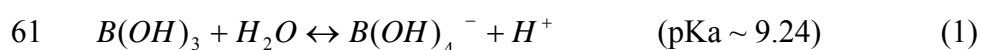
42 **1. Introduction**

43

44 Boron is being used in antifungal and detergent formulations, bleaching agents, glass
45 and ceramics, shielding in nuclear reactors, semiconductors, high-hardness engineering
46 materials etc.¹. Because of such wide spread applications of boron, its consumption in
47 the world is kept on increasing. This creates the problem of the appearance of
48 significant quantity of boron in the residual solutions originating from manufacturing
49 and disposal of boron containing materials that can contaminate ground water streams
50 which are in domestic and agricultural applications^{2,3}. Boron is termed as micronutrient

51 for all living organisms but its little excess consumption can be toxic⁴⁻⁶. For humans,
52 boron can represent reproductive dangers and has suspected teratogenic properties. The
53 recommended limits for boron in drinking water are 0.5 and 1.0 mg/L by World Health
54 Organization (WHO) and the European Union, respectively^{7,8}. A major limiting factor
55 of boron is its possible damage to plants and crops. Amongst the more sensitive crops
56 are citrus trees where damage is noticeable when boron level is more than 0.3 mg/L in
57 the irrigation water⁹. Hence it is an important issue to monitor and remove excess
58 (above stipulated limit) boron from water. Boron is usually present in water as boric
59 acid which is a Lewis acid as it abstract OH⁻ ion from water and release H⁺ ion.

60 This chemical equilibrium can be written as¹⁰:



62 The presence of boron in different molecular species has great effect on the efficient
63 removal or extraction of boron from the aqueous solutions. The changing concentrations
64 of boron species in aqueous solutions at different pH calculated by Chemical
65 equilibrium software MEDUSA¹¹ are shown in Figure 1. Adsorption^{12,13},
66 electrocoagulation¹⁴, ion exchange^{15,16}, reverse osmosis^{17,18} which are very common
67 processes for water treatment can remove boron efficiently when it is in the ionic form
68 whereas solvent extraction technique can remove boron in its neutral form. Organic
69 molecules with the functional group 1,3 diol form 1:1 complex with an undissociated
70 boric acid and that allow its extraction into non-aqueous solvents^{19,20}. Hence referring to
71 Figure 1, solvent extraction or solvent extraction based liquid membrane appears to be a
72 potential technique for the treatment of water of natural origin which is showing pH
73 near 7. Solvent extraction of boron using 1,3 diols has been reported in different
74 analytical^{21,22} and industrial methods of boron recovery²³⁻²⁶. The relative extraction
75 efficiency of different diols for boron depends on their molecular and structural

76 geometry^{27,28}. The main constraint observed in the utilization of diols as extractants is
77 their significant solubility in the aqueous solution. In present work, efficiency of
78 different modifiers to hold diol molecules in the organic phase (Kerosene or Solveso
79 200) is investigated. Based on the information available in the literature, extractants 2-
80 methyl-2,4- pentanediol (MPD), 2-ethyl-1,3 hexanediol (EHD), 2,2,4-trimethyl-1,3-
81 pentanediol (TMPD) and 2-butyl-2-ethyl-1,3-propanediol (BEPD) and modifiers
82 decanol, 2,6-dimethyl-4-heptanone (DMH) and tributyl phosphate (TBP) are selected to
83 study the liquid-liquid extraction of boron from neutral aqueous solutions. The objective
84 of this study is to obtain a suitable solvent extraction method for the removal of boron
85 from environment oriented solutions such as sea water and various boron carrying
86 industrial waste solutions as spent cutting fluid and phosphate passivation bath.

87

88 **2. Experimental**

89

90 2.1 Reagents

91

92 MPD, EHD, TMPD and BEPD were purchased from Aldrich Chemicals. Boric acid,
93 decanol, and TBP were obtained from MERCK. Kerosene procured from Fluka
94 Analytical and Solveso 200 was kindly donated by Exxon Mobil Chemical, Spain.
95 These organic chemicals were used as received. Azomethine H, ascorbic acid,
96 ammonium acetate and acetic acid obtained from Panreac were used for the
97 spectrophotometric analysis of boron. Other reagents used were of analytical reagent
98 grade.

99 Required concentration of boron solutions were prepared by dissolving appropriate
100 quantity of boric acid in distilled water containing 0.5 M NaCl. The salt content in the

101 solutions of boron was maintained for better phase separation and considering potential
102 use of this method for extractive separation of boron from solutions containing
103 significant quantity of salt such as sea water and industrial waste solutions.

104 Two industrial waste solutions namely spent cutting fluid and phosphate passivation
105 bath were obtained from Tratamiento y Recuperaciones Insustriales S.A., Spain. Both of
106 these solutions were first treated to separate out their organic component. The aqueous
107 part of spent cutting fluid and phosphate passivation bath contained about 750 and 150
108 mg/L of boron respectively.

109

110 2.2 Extraction procedure

111

112 Ten mL of aqueous phase containing 1 g/L (or specified concentration) of boron was
113 equilibrated with an equal volume organic phase using a horizontal mechanical shaker
114 (SBS Mechanical Shaker) at 140 rpm for 20 min at room temperature ($23\pm 3^\circ\text{C}$).

115 According to preliminary experiments this time of shaking is enough to reach the
116 equilibrium. After extraction, two phases were allowed to separate and the aqueous
117 phase was analysed for the concentration of boron. The concentration of boron in the

118 loaded organic phase was determined by mass balance of the concentrations of boron in
119 the aqueous phase before and after extraction. This mass balance was further confirmed
120 analysing boron in the stripped aqueous phase. For stripping experiments boron loaded

121 organic phase was equilibrated with equal volume of NaOH or KOH solution for 20
122 min. A complete stripping of boron was assured by equilibrating organic phase
123 repeatedly with stripping solution till the pH of the equilibrated strip solution was >12 .

124

125 2.3 Determination of boron

126

127 Boron in the aqueous phase was analysed by spectrophotometry using Azomethine H as
128 chromogenic reagent²⁹. A suitable aliquot (containing ~ 20 µg of boron) was added to a
129 25 mL flask containing 5 mL of ammonium acetate buffer solution of pH 4.5. To this, 5
130 mL of chromogenic reagent (10g/L Azomethine H + 20g/L ascorbic acid) was added
131 and solution was made-up to 25 mL with double distilled water. The optical density of
132 this coloured solution was measured after 2 h at 415 nm on a Shimadzu UV-1603
133 UV-visible spectrophotometer.

134 The extraction of boron is reported as percentage extraction (%E) which was calculated
135 as:

$$136 \quad \%E = \frac{[B]_{ini} - [B]_{eq}}{[B]_{ini}} \times 100 \quad (2)$$

137 where $[B]_{ini}$ and $[B]_{eq}$ are the concentrations of boron in the aqueous phase, before and
138 after equilibration with the organic phase respectively. The results reported are within
139 $\pm 3\%$ accuracy and $> 95\%$ mass balance.

140

141 2.4 Determination of dissolved BEPD and TMPD in the aqueous solutions

142

143 Aqueous samples were analysed for dissolved extractant on Shimadzu GC-2010Plus
144 chromatograph with a TRB1 (30 m x 0.25 mm x 0.5 µm) capillary column which was
145 maintained at 174°C. For the calibration studies, standard solutions of weighed quantity
146 of BEPD and TMPD were prepared in methanol/water mixture (50% v/v). In separate
147 analysis the retention times noted for BEPD and TMPD were 2.54 min and 1.67 min
148 respectively. Through auto sampler unit, 0.6 µL of sample solution was injected in the
149 injection port of gas chromatograph system. The split injection mode with split ratio

150 1:25 was used for these analyses. Concentrations of diols were calculated by peak area
151 integration method.

152

153

154 **3. Results and discussion**

155

156 3.1 Effect of solvent modifiers

157

158 Organic diols show good solubility in polar and polarisable aromatic solvents; whereas
159 non polar aliphatic ones need some polar additives to dissolve diol molecules. Two
160 different kind of commercial diluents, aromatic Solvesso 200 and aliphatic Kerosene
161 were selected to study the extraction of boron by different diols. The comparative
162 extraction data are presented in Figure 2. Due to very less solubility of selected diols in
163 Kerosene, extraction of boron could not be carried out using only Kerosene as a diluent.
164 Diols are sufficiently soluble in Solvesso 200 and it can be used as diluent for the
165 extraction of boron without any solvent modifier. In case of Solvesso 200 as diluent,
166 extraction is improved with the addition of decanol in the system. This increase in the
167 extraction of boron may be due to increase in the net polarity of the organic phase by
168 coupling of decanol with naphthenic molecules. To make a solution of required
169 concentrations of diol compound in Kerosene, a sufficient quantity of solvent modifier
170 is required to be incorporated in the organic phase. In Kerosene medium, the extraction
171 of boron decreases with the addition of selected solvent modifiers. Molecules of solvent
172 modifiers are least reactive towards aliphatic components of Kerosene but they show
173 interaction with diols in terms of hydrogen bonding. Significantly high antagonistic
174 effect on the extraction of boron is observed when TBP was used as a modifier with all

175 diols studied. Looking into the extraction capacities of different diols for boron, MPD
176 appears to be very weak extractant. The results presented in Figure 2 are for single stage
177 extraction of boron and the envisage problem of extractant solubility in aqueous phase
178 is not reflected in these data. In further investigations EHD, TMPD and BEPD are used
179 as extractants with solvent modifiers; decanol and DMH. In case of Kerosene
180 containing 10% decanol as a diluent, the extraction of boron is sufficiently close to that
181 obtained with Solvesso 200. Hence besides good extractability of boron in Solvesso
182 200, to avoid of using a diluent of hazardous aromatic nature (high vapour pressure, low
183 flash point and hazardous to handle), decanol-Kerosene mixture was chosen as a diluent
184 for the rest of the studies.

185

186 3.2 Effect of successive extractions

187

188 For large scale or continuous extractions such as counter current mixer-settler process or
189 liquid membrane pertractions, a stability of extractant in the organic phase has utmost
190 importance. Results depicted in Figure 3a, b and c show the percentage extraction of
191 boron in the organic phase obtained by EHD, TMPD and BEPD with different quantity
192 of decanol in the successive experiments of liquid-liquid equilibrium. In the successive
193 experiments, a single extraction step involved extraction of boron in the organic phase,
194 the stripping into alkaline aqueous solution and finally washing of organic phase with
195 acidified aqueous solution to remove alkaline contamination of the stripping solution. In
196 the first step, extraction of boron is more when EHD and TMPD are in the organic
197 phase compared to that with BEPD but in the successive extractions, the organic phase
198 containing EHD and TMPD could not maintain their efficiency for boron extraction.
199 This is apparently due to the losses of extractant in the aqueous solutions. In case of

200 BEPD, successive extractions showed comparatively better consistency in the extraction
201 of boron. At 10% of decanol in the organic phase, extractions of boron with EHD and
202 TMPD are 85% and 75% respectively but after 4 successive extractions, in the fifth step
203 the same quantities are reduced to 8% and 9% respectively. Whereas in case of BEPD
204 comparatively less, i.e. about 12% decrease in the extraction of boron is noted after four
205 successive extractions. As shown in Figure 3c, 50% of decanol with BEPD in the
206 organic phase is enough to keep solvent extractability in successive runs. The stronger
207 interaction of decanol makes BEPD to retain more in the organic phase but at the same
208 time it reduces the efficiency of BEPD to extract boron. Based on the higher efficiency
209 for the extraction of boron by TMPD and better stability of BEPD in the organic phase,
210 further studies are continued with these two diols. Similar results are seen (Fig. 4a and
211 b) when DMH is used as solvent modifier but it is less effective than decanol to retain
212 diol molecules in the Kerosene phase.

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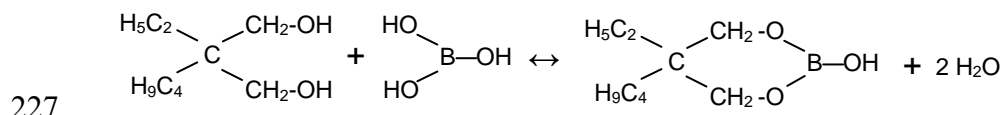
214 3.3 Effect of the concentration of extractant

215

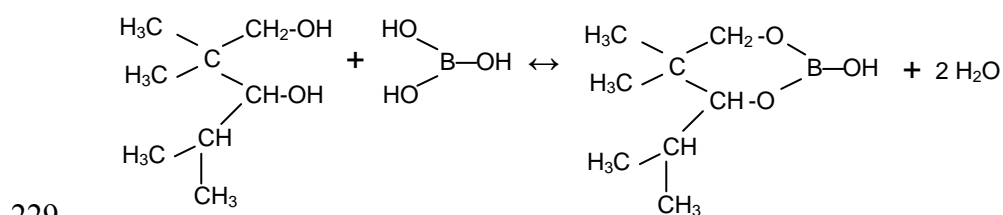
216 Figure 5a and b show the percentage extraction of boron with different concentrations
217 of TMPD and BEPD respectively in the organic phase. Extractions are carried out with
218 two different concentrations of decanol in the organic phase. The extraction of boron
219 increases with the concentration of diols in the organic phase. Extraction of boron is less
220 when organic phase contains higher concentration of decanol. This effect is more
221 pronounced for BEPD than for TMPD. More than 90% of boron could be extracted
222 using 0.5 M TMPD in the organic phase whereas 73% of boron is getting extracted with
223 similar concentration of BEPD in the organic phase. As explained in the literature,^{20, 23}

224 and also based on our results of liquid-liquid extraction, boron forms diesters when
 225 reacting with diols. Equilibria of boron with BEPD and TMPD can be written as:

226



228



231 A certain percentage of the extraction of boron in absence of extractant is also noted
 232 which is due to some weak complexing of boron with decanol in the organic phase.
 233 Extraction of boron by higher molecular weight alcohols is also reported in the
 234 literature³⁰.

235

236 3.4 Effect of the concentration of decanol

237

238 As suggested by Turner and Williamson³⁰ and also based on the data presented in
 239 Figure 6 boron forms weak complex with decanol which is resulting its extraction into
 240 the organic phase. Extraction of boron increases with the concentration of decanol in the
 241 organic phase. Maximum about 16% extraction of boron could be achieved using 100%
 242 decanol in the organic phase. Looking into Figure 6, boron is also having some
 243 solubility in Kerosene. About 3% extraction of boron is noted in Kerosene which is
 244 confirmed by repeat experiments.

245 The effect of the varying concentrations of decanol at constant concentration of
246 extractant in organic phase on the extraction of boron is shown in Figure 7. Extraction
247 of boron decreases with increasing concentration of decanol in the organic phase. This
248 is probably due to the hydrogen bonding interaction of decanol with extractant
249 molecules which masks its reactive efficiency for boron. Decrease in the extraction of
250 boron is about 8% more with BEPD than TMPD and it suggests greater interaction of
251 decanol with BEPD than with TMPD. This is also reflected in the higher retention of
252 BEPD by decanol in the organic phase during successive steps of extractions (Fig. 3b
253 and 3c).

254 Further the quantitative analysis of the solubility of BEPD in the aqueous phase is done
255 using gas chromatographic analysis. A method for the measurement of diols in the
256 aqueous solutions was developed using direct sample injection method. An optimization
257 and calibration studies showed $\pm 5\%$ accuracy in the determination of dissolved diols.
258 The concentrations of BEPD and TMPD in the aqueous solutions obtained after
259 equilibration with organic phase containing different concentrations of decanol are
260 presented in Figure 8a and 8b. Retention of BEPD in the organic phase increases with
261 the addition of polar solvent modifier, decanol in the system. This behavior is more
262 marked at high concentration of BEPD. On the other hand, the influence of decanol
263 with TMPD is comparatively weak. The effect of the aqueous solubility of both
264 extractants is in concordance with the decrease in the efficiency of boron extraction in
265 the successive extraction experiments (Fig. 3b, and 3c). In case of TMPD, extraction of
266 boron was decreased by 75% after four successive extractions whereas under similar
267 conditions, extraction of boron was decreased by 44% with BEPD.

268

269 3.5 Effect of the aqueous phase pH

270

271 As given in Eq. (3) and (4), TMPD and BEPD react with undissociated molecule of
272 boric acid. Hence prime requirement for obtaining better extraction of boron is its
273 availability in the form of H_3BO_3 in the aqueous solution. Referring to Eq.1 and Fig.1,
274 the concentration of undissociated H_3BO_3 in the aqueous solution depends on its pH.
275 Results obtained for the extraction of boron at different pH are shown in Figure 9a.
276 Extraction of boron remains fairly constant up to pH 7 and beyond pH 8 decreases
277 sharply. Boron is practically non extractable above pH 11. This condition of aqueous
278 phase is favorable for the stripping of boron from the organic phase. About 90% boron
279 could be extracted by 0.3 M TMPD in 30% decanol/Kerosene phase from aqueous
280 solutions of pH less than 7 and above this pH extraction of boron dropped down to 80%
281 at pH 8 and 52% at pH 9.4. Similar tendency is obtained with BEPD. As shown in Eq.1,
282 boric acid is a Lewis acid and its extraction from aqueous phase may change pH of the
283 equilibrated aqueous phase. To observe this effect pH of the equilibrated aqueous phase
284 solutions are measured (Fig. 9b). Practically no change in the equilibrated aqueous
285 phase pH is seen to that of its initial value. This can be explained as, at lower pH
286 majority of boric acid is present in undissociated form and hence it is not contributing to
287 the pH of the aqueous phase.

288

289 3.6 Extraction isotherms

290

291 Extractions of boron are studied from different concentrations of boron in the aqueous
292 phase by both TMPD and BEPD. As shown in Figure 10a and b, TMPD exhibit greater
293 capacity for the loading of boron into the organic phase compared to BEPD. With 0.3 M
294 TMPD in presence of 75% of decanol, maximum around 3500 mg/L of boron could be

295 loaded into organic phase where as with similar concentrations of BEPD and 50%
 296 decanol, nearly 2450 mg/L of boron could be loaded into the organic phase. Information
 297 depicted in Figure 10a and b is useful for selecting the concentration of extractant or the
 298 ratio aqueous/organic phase as per the concentration of boron to be removed or recover
 299 from aqueous feed solution. These isotherms are also useful for planning multi-step
 300 mixer-settler experiments.

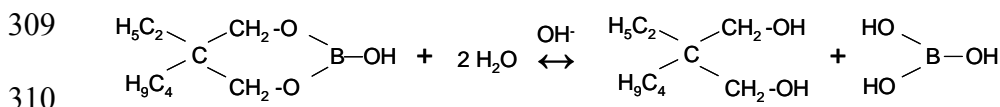
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302 3.7 Stripping of boron from loaded organic phase

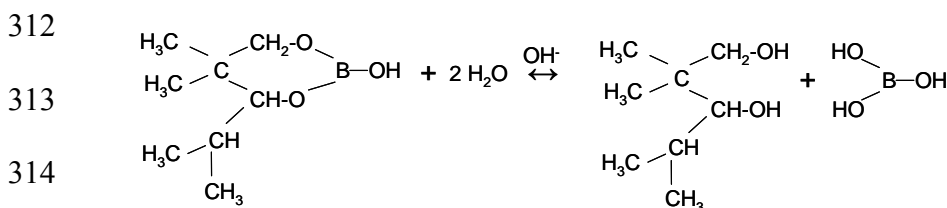
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304 As shown in Figure 9a, boron is practically non extractable beyond pH 11. This can be
 305 referring to the presence of boron totally in the form of borate (Fig. 1). Taking of this
 306 advantage, stripping of boron are carried out using alkaline solutions. Stripping of boron
 307 can be explained with following chemical equations:

308

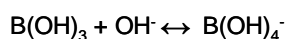


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316



318

For the preliminary experiments, stripping solution used was 0.2 M NaOH + 0.3 M

319

NaCl when worked with lower concentrations of boron but for higher concentrations of

320 boron, more concentrated NaOH or multi-step stripping was needed. This can be
321 explained with data given in Table 1. A quantitative single step (volume ratio 1:1)
322 stripping of boron could be obtain using 0.2 M NaOH + 0.3 M NaCl from loaded
323 organic containing up to 2000 mg/L of boron. Along with NaOH, KOH solutions are
324 also studied for the stripping of boron. With NaOH, sodium tetraborate of molecular
325 formula $\text{Na}_2\text{B}_4\text{O}_7$ is formed which is having 60 g/L of aqueous solubility. Where as with
326 KOH, potassium tetraborate ($\text{K}_2\text{B}_4\text{O}_7$) is formed which is having aqueous solubility
327 around 170 g/L. For commercial applications where higher quantity of boron is to be
328 recovered, KOH is the preferred stripping agent because of the higher aqueous
329 solubility of $\text{K}_2\text{B}_4\text{O}_7$ and further for its easy crystalline recovery.

330

331 3.8 Extraction of boron from industrial waste solutions

332

333 Extraction of boron was studied from two industrial aqueous waste solutions derived
334 from spent phosphate passivation bath and cutting fluid. The organic additives of
335 surfactant quality, present in these solutions created problem of phase separation in
336 liquid-liquid extraction experiments. Hence a pre-separation of organic from these
337 solutions became imperative. In case of spent cutting fluid a standard method of phase
338 inversion by the addition of sulphuric acid was applied to separate organic component
339 from the bulk aqueous solution. For spent phosphate passivation bath different methods
340 were tested to remove organic from the homogeneous solution. It was observed that
341 majority of organic matter is getting separated by the addition of group I and II metal
342 chloride salts and then keeping the mixture for long standing. The other method
343 successfully tried was ultra filtration of this solution. In this case the solution was
344 passed through a 10 kDa regenerated cellulose membrane (Millipore YM10). In both of

345 these methods the volume of the organic separated was about 8% of the original volume
346 of the spent passivation bath solution. The data pertaining to the extraction of boron
347 from industrial waste solutions is described in Table 2. In single step experiment about
348 72% of boron was removed when 0.6 M BEPD in 25% decanol/Kerosene was used
349 whereas in similar experiment, >93% of boron was eliminated by 0.6 M TMPD in 25%
350 decanol/Kerosene.

351

352 **Conclusions**

353

354 Among three modifiers studies, TBP exhibited maximum antagonistic effect towards
355 the extraction of boron without imparting any significant benefit for diols stability in the
356 organic phase. Compared to other modifiers studied, decanol gives better stability to
357 diol molecules in the Kerosene phase and exhibit lesser negative effect on the extraction
358 of boron. Next to decanol a low viscous DMH can be used as solvent modifier.
359 Extraction efficiency for boron is better with TMPD than BEPD but in the successive
360 extractions, better consistency in the extractions of boron is obtained with BEPD in
361 combination with decanol in the organic phase. The gas chromatographic analyses
362 indicating solvent losses in the aqueous phase at different concentrations of decanol are
363 given to support the solvent extraction data. Looking into extraction efficiency and their
364 stability in the organic phase, TMPD and BEPD are potential extractants to remove
365 boron from neutral or acidic aqueous solutions. An efficient and selective extraction of
366 boron from industrial waste solutions such as spent phosphate passivation bath and
367 cutting fluid can be achieved using 0.6 M BEPD and 0.6 M TMPD in 25%
368 decanol/Kerosene medium.

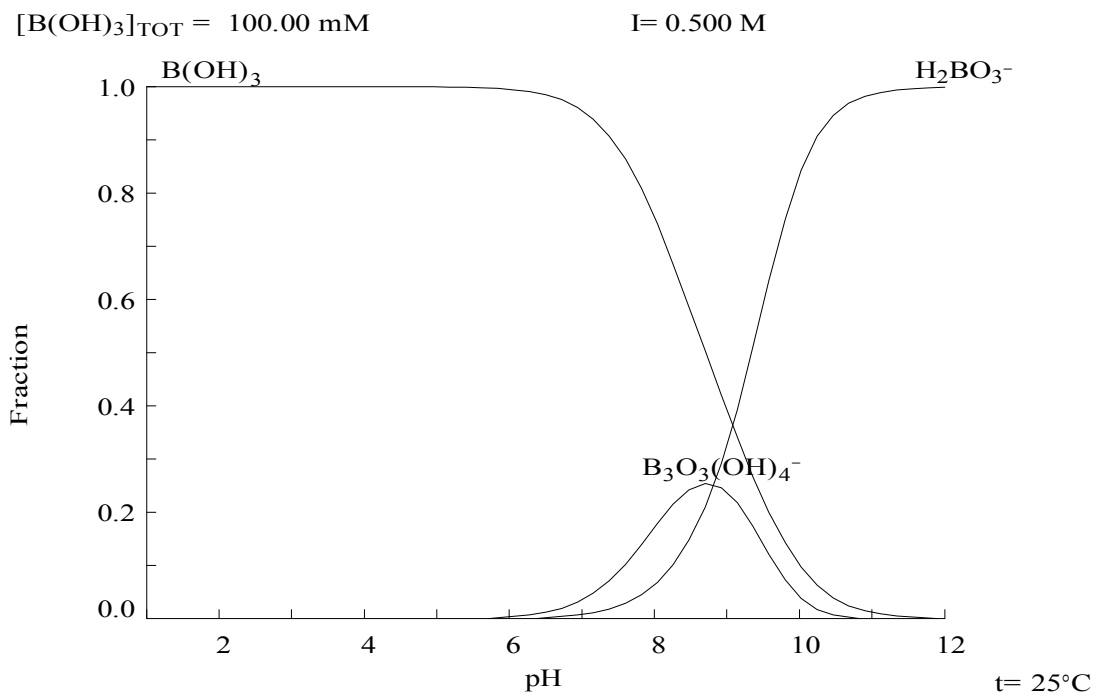
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370 **Acknowledgments**

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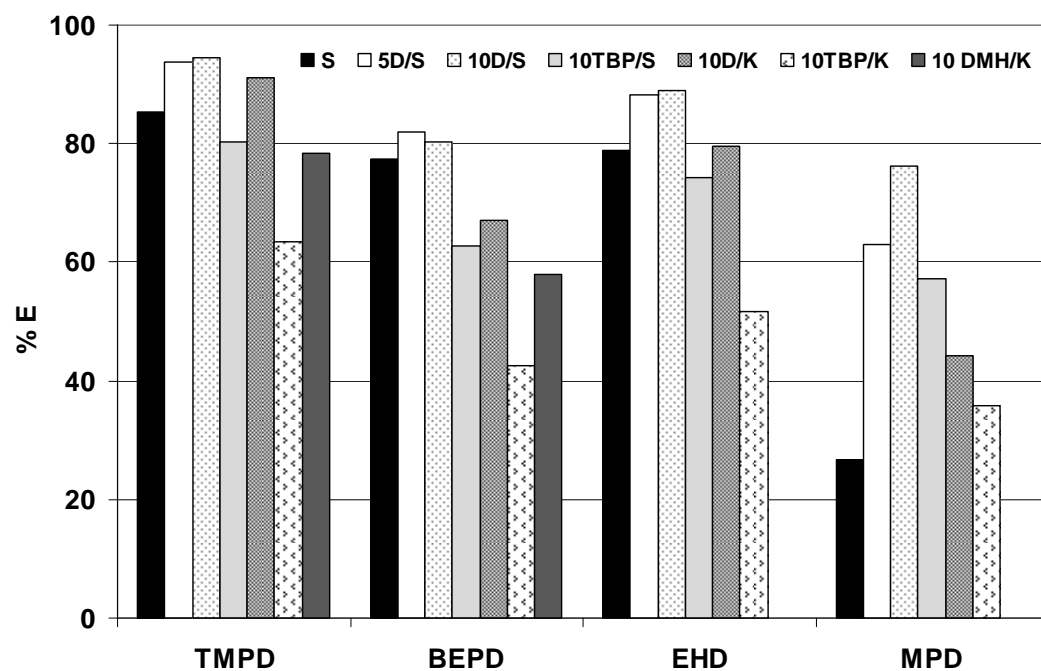
372 This work was supported by the Spanish Ministry of Science and Innovation with its
373 grant no. CTQ2008-00417/PPQ. C.S. Kedari acknowledges the financial support of the
374 Spanish Ministry of Science and Innovation (Project: SAB2009-0143). We also thank to
375 TRISA (Tratamiento y Recuperaciones Industriales S.A.) the supply of industrial
376 wastewaters containing boron.

377



378

379 **Figure 1.** Distribution of boron species in the aqueous phase at different pH
 380



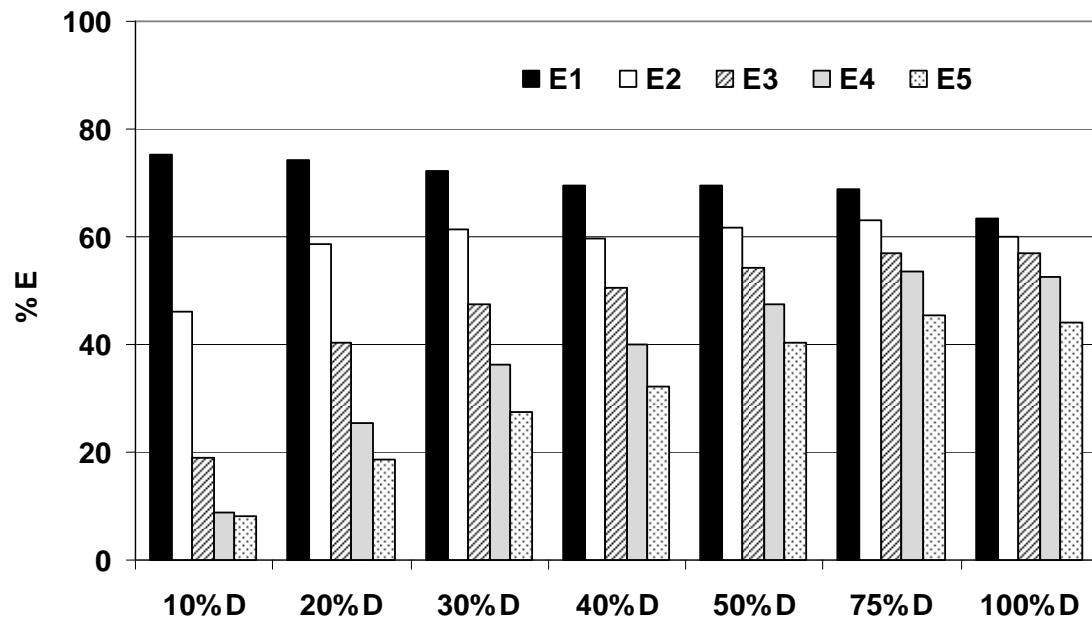
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382 **Figure 2.** Effect of solvent modifiers on the boron extraction by 0.265 M of MPD,

383 EHD, TMPD and BEPD. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

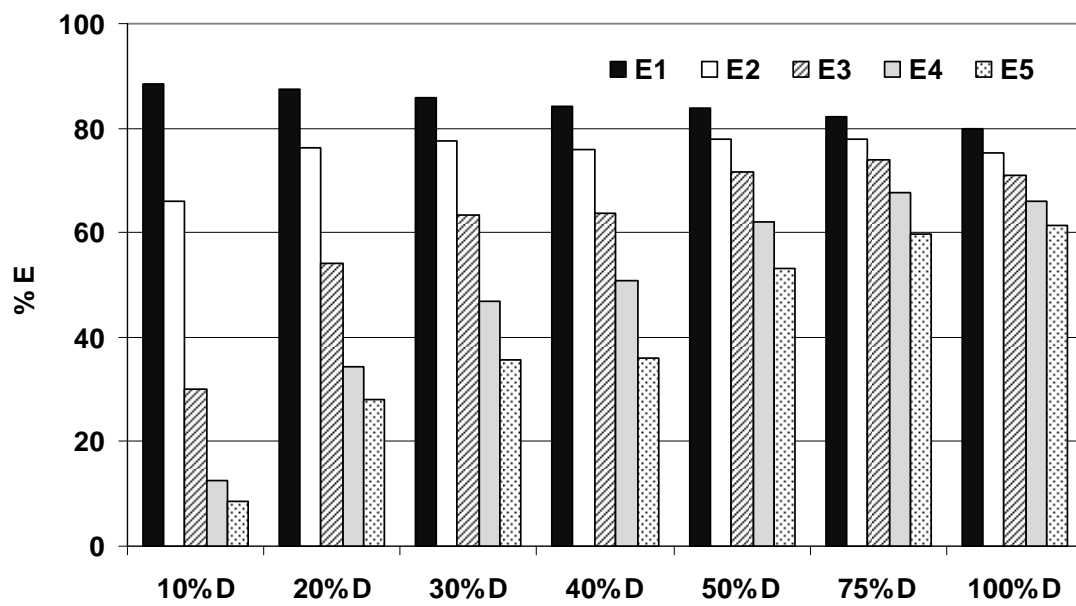
384 (Solvesso “S”, Kerosene “K” and Decanol “D”)

385



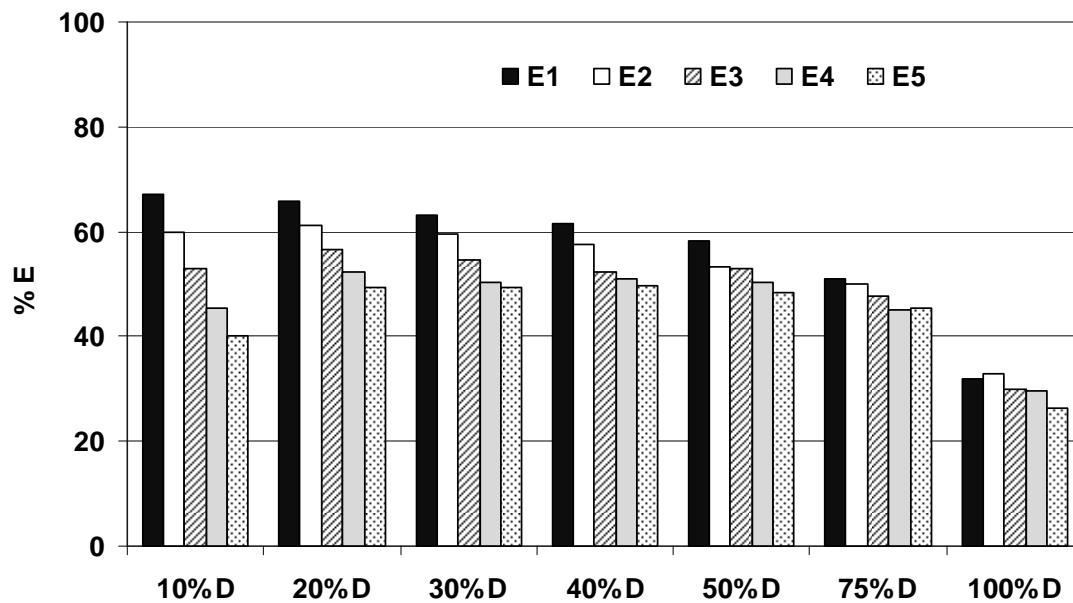
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387 Figure 3a



388

389 Figure 3b

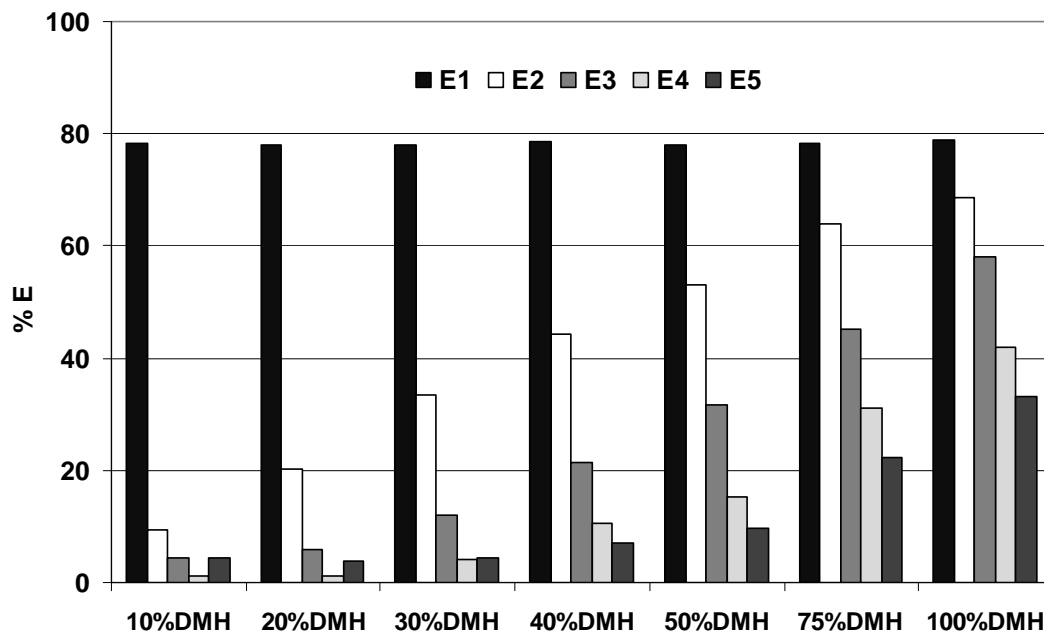


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391 Figure 3c

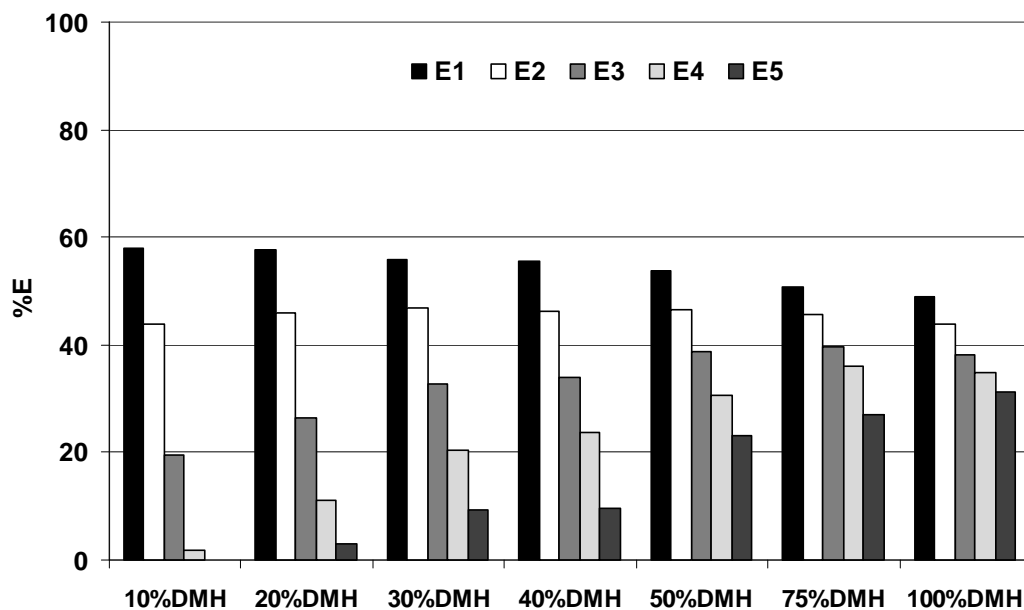
392 **Figure 3.** Effect of five successive extractions on the percentage extraction of boron by
 393 0.265 M (a) EHD, (b) TMPD and (c) BEPD for different concentrations of
 394 decanol in Kerosene. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

395



396

397 Figure 4a



398

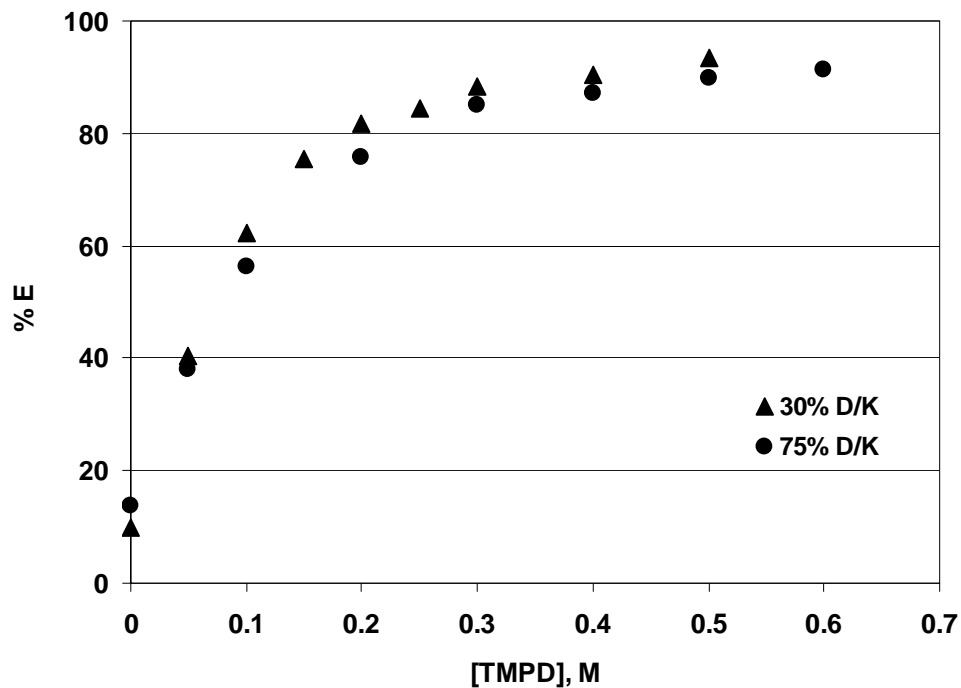
399 Figure 4b

400 **Figure 4.** Effect of five successive extractions on the percentage extraction of boron by

401 0.265 M (a) TMPD and (b) BEPD for different concentrations of DMH in

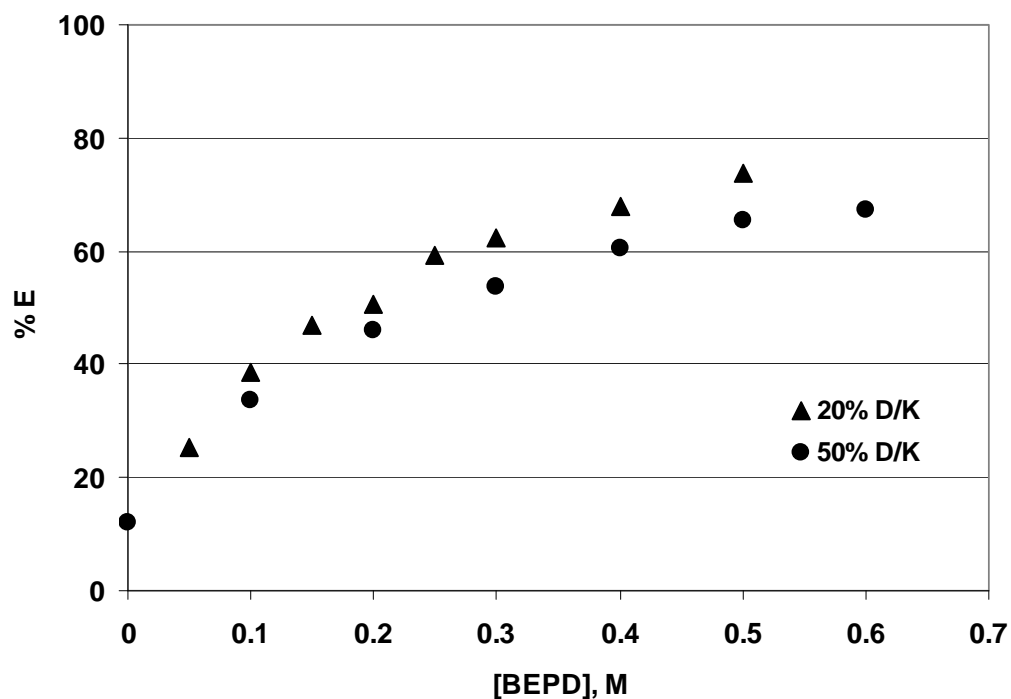
402 Kerosene. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

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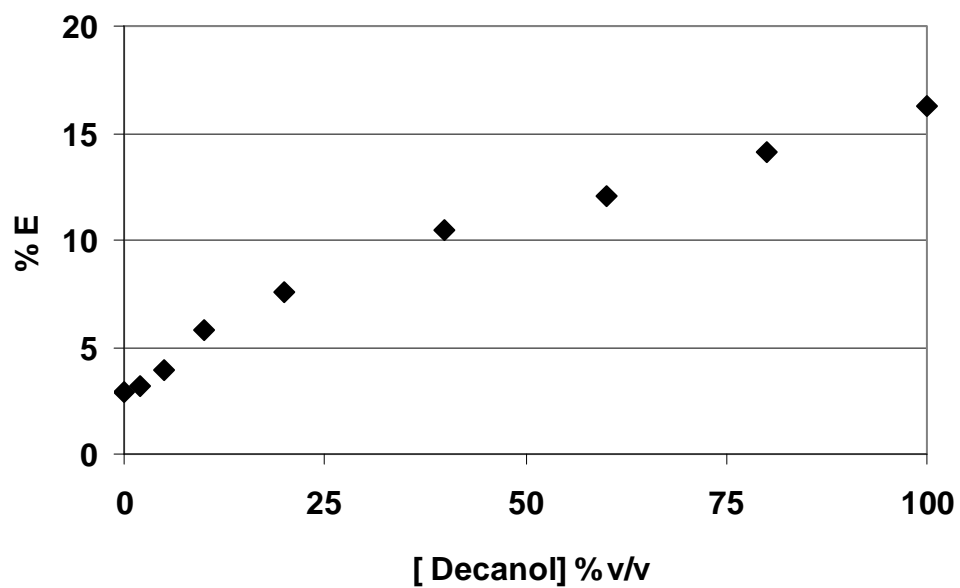
405 Figure 5a



406

407 Figure 5b

408 **Figure 5.** Extraction of boron at different concentration of (a) TMPD and (b) BEPD in
 409 the organic phase. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

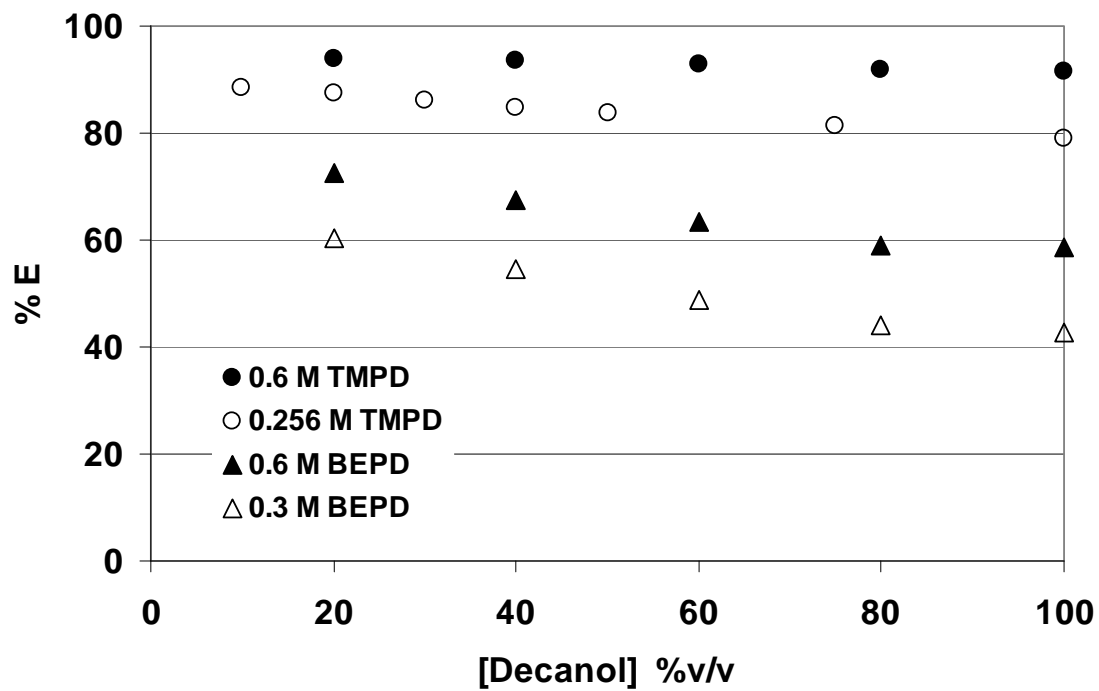


411

412 **Figure 6.** Extraction of boron by varying concentration of decanol in the organic phase.

413 Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

414

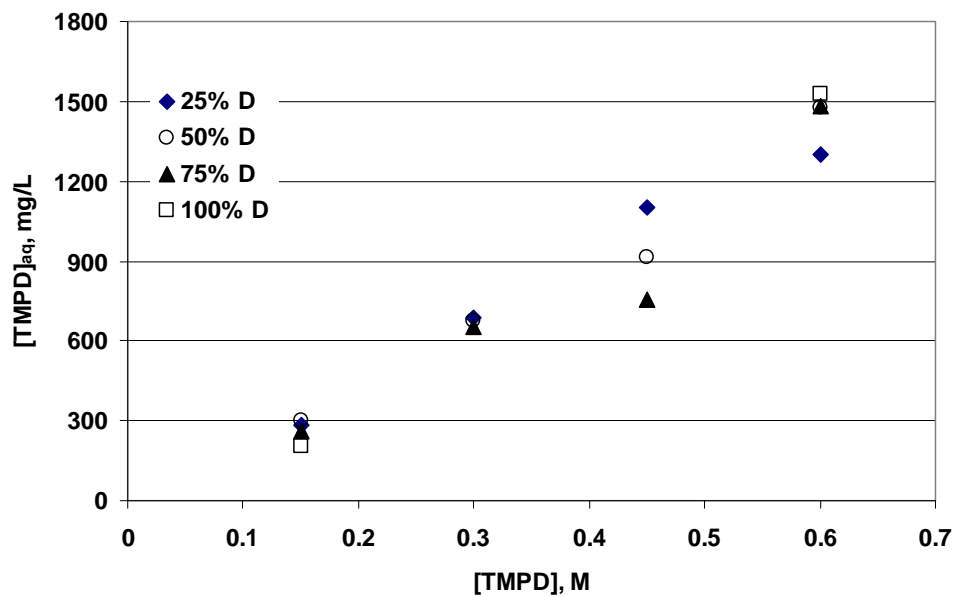


415

416 **Figure 7.** Effect of the concentration of decanol on the extraction of boron in Kerosene

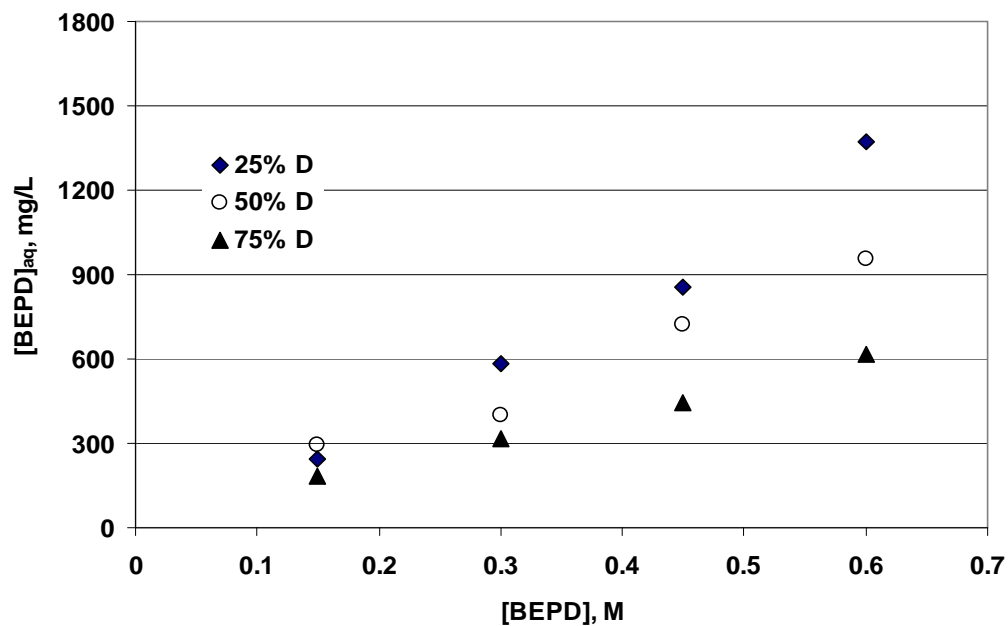
417 by TMPD and BEPD. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

418



419

420 Figure 8a

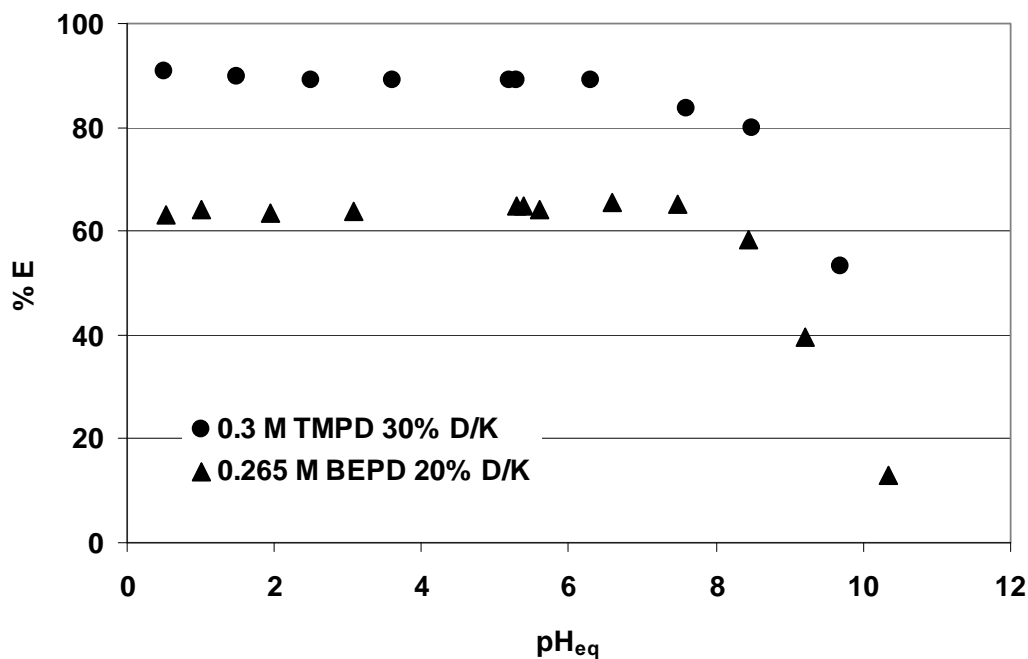


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422 Figure 8b

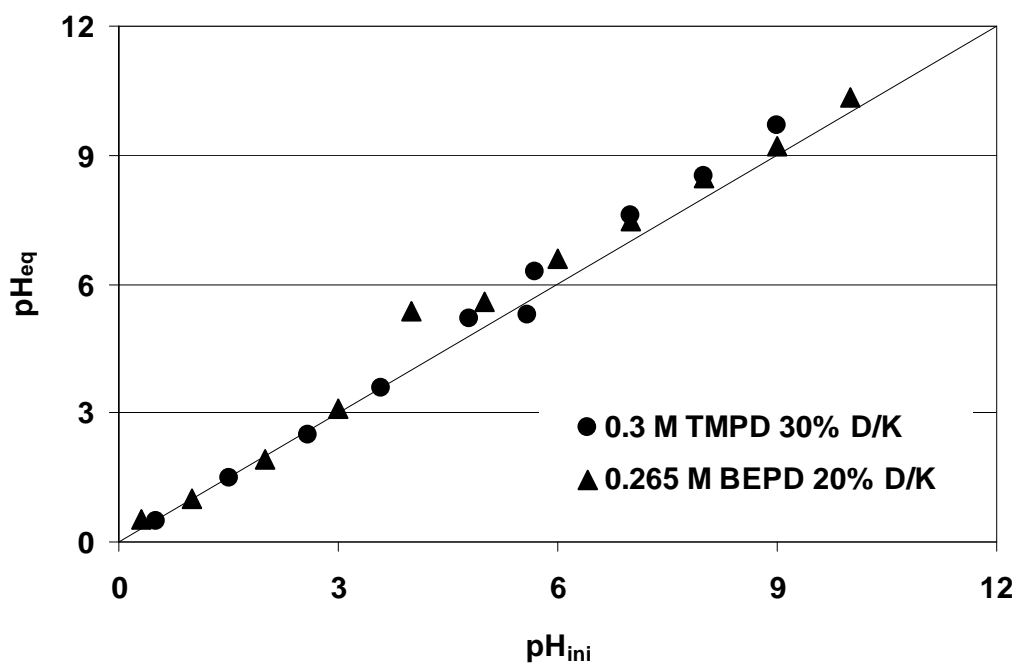
423 **Figure 8.** Extractant solubilized (a) TMPD and (b) BEPD in the equilibrated aqueous
 424 phase as a function of its nominal concentration in the organic phase. Aqueous
 425 phase: 1000 mg/L of Boron in 0.5M NaCl

426



427

428 Figure 9a



429

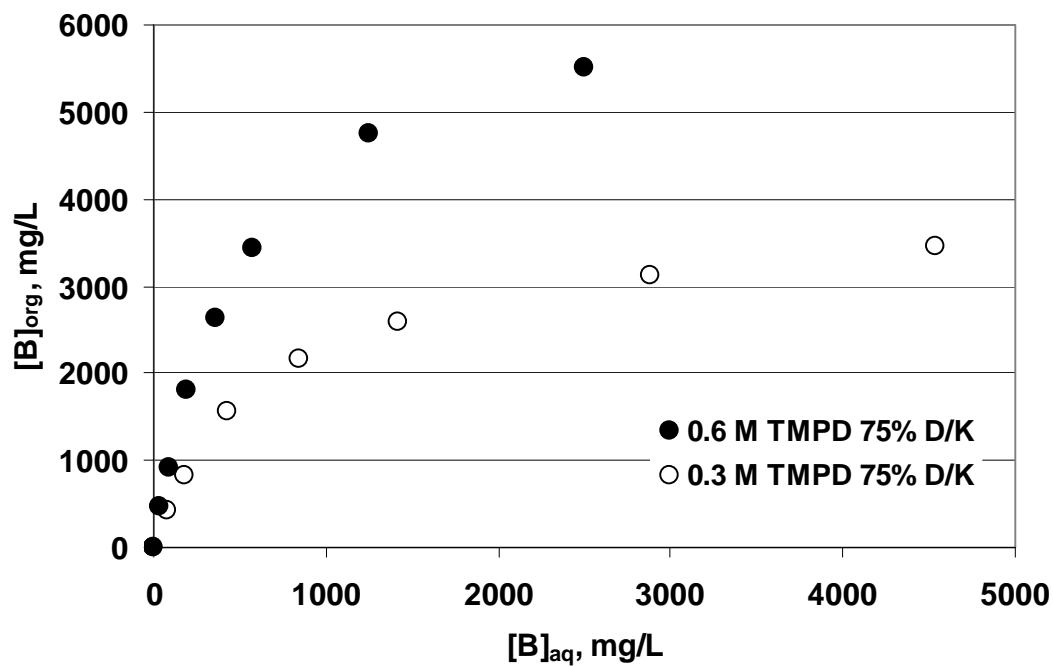
430 Figure 9b

431 **Figure 9.** (a) Effect of aqueous phase pH on the percentage extraction of boron by

432 TMPD and BEPD, and (b) variation of pH of the aqueous phase before and

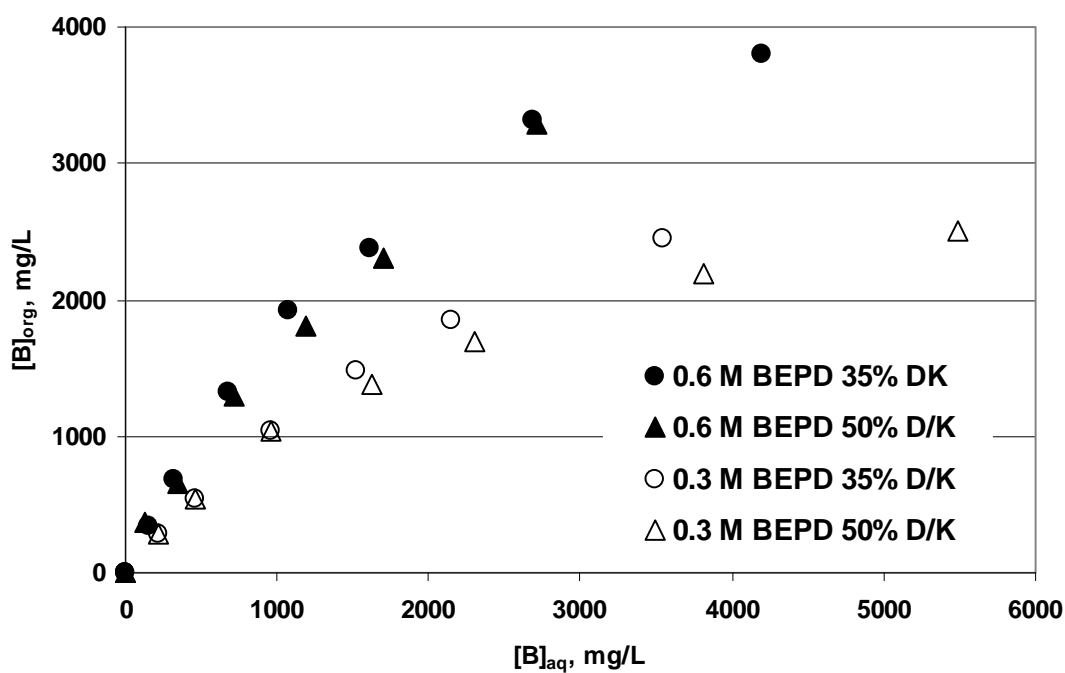
433 after equilibration. Aqueous phase: 1000 mg/L of Boron in 0.5M NaCl

434



435

436 Figure 10a



437

438 Figure 10b

439 **Figure 10.** Extraction isotherm of boron by (a) TMPD and (b) BEPD

440

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542

543 Table 1
 544
 545 Stripping of boron from loaded organic phase
 546

Organic phase	Stripping solution	[B] _{org} ^a mg/L	[B] _{aq} ^b mg/L	pH ^c	% Stripping
0.6 M BEPD + 25% decanol	0.1 M KOH + 0.2 M NaCl	725	659	11.4	91
	0.2 M KOH + 0.3 M NaCl	725	724	12.5	100
	0.2 M NaOH + 0.3 M NaCl	550	547	12.7	100
	0.2 M NaOH + 0.3 M NaCl	2122	1947	11.0	97
	0.2 M NaOH + 0.3 M NaCl	2689	2331	10.3	87
0.6 M TMPD + 25% decanol	0.1 M NaOH + 0.2 M NaCl	947	855	11.8	90
	0.2 M NaOH + 0.3 M NaCl	947	931	12.6	98
	0.1 M KOH + 0.2 M NaCl	947	852	11.2	90
	0.2 M KOH + 0.3 M NaCl	947	920	12.5	97

547 a, Initial concentration of boron in loaded organic phase
 548 b, Concentration of boron in the aqueous strip phase after equilibration
 549 c, Aqueous phase pH after equilibration
 550

551 Table 2
 552
 553 Extraction of boron from industrial spent solutions
 554

Extractant	Industrial Solutions	[B] _{initial} mg/L	[B] _{final} mg/L	Recovery %
1. Spent phosphate passivation bath preconditioned* to				
0.6 M BEPD + 25% decanol in Kerosene	a. 0.6 M of NaCl	162	45	72
	b. 0.4 M of MgCl ₂	148	21	85
	c. Passed through 10 kDa UF membrane	326	90	72
0.6 M TMPD + 25% decanol in Kerosene	a. 0.6 M of NaCl	162	11	93
	b. 0.4 M of MgCl ₂	148	10	93
	c. Passed through 10 kDa UF membrane	326	20	94
2. Spent cutting fluid preconditioned* to				
0.6 M BEPD + 25% decanol in Kerosene	a. 2% H ₂ SO ₄	748	195	74
0.6 M TMPD + 25% decanol in Kerosene	b. 2% H ₂ SO ₄	748	47	94

555 *, Composition of waste solution was modified to separate out its organic component
 556