Characteristic Sorption of \( \text{H}_3\text{BO}_3/\text{B(OH)}_4^- \) on Magnesium Oxide

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The reaction mechanism of \( \text{H}_3\text{BO}_3/\text{B(OH)}_4^- \) with MgO in aqueous phase was investigated using sorption isotherm, XRD, \(^{11}\text{B}\)-NMR and FTIR. Release of Mg\(^{2+}\) was observed soon after contact of MgO with \( \text{H}_3\text{BO}_3 \) and maximum released Mg\(^{2+}\) was proportional to the initial boron concentration, suggesting ligand-promoted dissolution of MgO by \( \text{H}_3\text{BO}_3 \). The molecular form of \( \text{H}_3\text{BO}_3 \) was more reactive with MgO in FTIR. Release of Mg\(^{2+}\) after sorption of \( \text{H}_3\text{BO}_3 \). The molar ratio of \([\text{B}]\) increased with \( \text{H}_3\text{BO}_3 \) sorption density. XRD patterns for the solid residues were assigned to Mg(OH)_2 and peaks broadened with increasing \( \text{H}_3\text{BO}_3 \) sorption density, except for \((hkl)\) planes due to c-axis lattice strain induced by incorporation of \( \text{H}_3\text{BO}_3 \) between layers. These results indicated that \( \text{H}_3\text{BO}_3 \) interfered in the c-axis stacking of in Mg(OH)_2. Molecular \( \text{H}_3\text{BO}_3 \) acted as a trigger when reacting with the MgO surface, releasing Mg\(^{2+}\) to produce an unstable complex leading to the precipitation formation of Mg(OH)_2, which is a sink for the immobilization of \( \text{H}_3\text{BO}_3/\text{B(OH)}_4^- \). [doi:10.2320/matertrans.M-M2013814]

(Received April 13, 2013; Accepted June 18, 2013; Published August 2, 2013)

**Keywords**: trigonal boron, magnesium oxide, sorption mechanism, ligand-promoted dissolution, \(^{11}\text{B}\)-NMR

1. Introduction

Boron is a ubiquitous element in rocks, soil and water. Most of the Earth’s soils have less than 10 mg/kg boron, with high concentrations found in parts of the western United States and in other sites stretching from the Mediterranean to Kazakhstan. Soils typically have boron concentrations of 2 to 100 mg/kg, but the average concentration is 10 to 20 mg/kg, with large areas of the world being boron deficient. Boron concentrations in rocks range from 5 mg/kg in basalts to 100 mg/kg in shales, and averages 10 mg/kg in the Earth’s crust overall. Seawater contains an average of 4.6 mg/L boron with a range of 0.5 to 9.6 mg/L. Freshwater concentrations normally range from 0 to 1.5 mg/L, with higher concentrations found near regions of high soil boron levels.\(^1\)

Boric acid and boron salts have extensive industrial use in the manufacture of glass and porcelain, in wire drawing, the production of leather, carpets, cosmetics and photographic chemicals, for fireproofing fabrics and weatherproofing wood. Boron compounds are used in certain fertilizers for the treatment of boron deficient soils. Boric acid, which has mild bactericidal and fungicidal properties, is used as a disinfectant and a food preservative. Borax is widely used in the welding and brazing of metals, and more recently, boron compounds have found applications in hand cleansing, high-energy fuels, cutting fluids and catalysts.\(^2,3\) Dissolution of boric acid from coal fly ash has also been an environmental issue for many years,\(^3-5\) and the chemical states of boron in coal fly ash have been investigated by \(^{11}\text{B}\)-NMR and TOF-SIMS because it may control leaching into the environment.\(^6,7\) Additionally, boric acid (\(^{10}\text{B}\)) has the important ability to absorb neutrons, known as “boron neutron capture”\(^8,9\) and is thus applied as an absorbent in nuclear reactors.

Findings from human and animal experiments have shown that boron is a dynamic trace element that can affect the metabolism or utilization of numerous substances involved in life processes including calcium, copper, magnesium, nitrogen, glucose, triglyceride, reactive oxygen and estrogen.\(^10\) Through these effects, boron can affect the function or composition of several body systems including the immune system, blood, brain and skeleton.\(^11,12\) Maximum concentration limits are regulated at 10 and 1 mg/L for industrial drainage and drinking water, respectively, by the World Health Organization.\(^13\)

At ambient pH, the dominant species of boron is undissociated boric acid (\(\text{H}_3\text{BO}_3\)). Boron is, therefore, difficult to immobilize at circumneutral pH values using electrostatic interactions. Boric acid dissociates to tetrahydroxyborate, with a \(pK_a\) of 9.23.\(^14\)

\[
\text{H}_3\text{BO}_3 + \text{H}_2\text{O} \rightleftharpoons \text{B(OH)}_4^- + \text{H}^+ \quad pK_a = 9.23 \quad (1)
\]

When B concentration is less than 25 mM, only the monomers \(\text{H}_3\text{BO}_3\) and \(\text{B(OH)}_4^-\) exist. Triboric acid is formed when B concentration is greater than 25 mM, with \(pK_a\) of 6.84\(^,14\)

\[
3\text{B(OH)}_3 \rightleftharpoons \text{B}_3\text{O}_3(\text{OH})_4^- + \text{H}^+ + 2\text{H}_2\text{O} \quad pK_a = 6.84 \quad (2)
\]

Triborate contains two trigonal B atoms (\(^{13}\text{B}\)) and one tetrahedral B atom (\(^{10}\text{B}\)) per molecule, and can be formed by the reaction of boric acid with tetrahydroxyborate:

\[
2\text{B(OH)}_3 + \text{B(OH)}_4^- \rightleftharpoons \text{B}_3\text{O}_3(\text{OH})_4^- + 3\text{H}_2\text{O} \quad K = 110 \quad (3)
\]

The current primary method of boron removal is by sorption on boron-selective resins, which have \(\alpha\)-methyl glucamine groups containing many hydroxyl groups that form complexes with borate.\(^15\) These resins are relatively expensive (~100 USD/L) and not suitable for large scale use, for example, as reactive materials for groundwater treatment. For selection of reactive materials for a practical scale treatment system, the target compound reactivity, permeability, accessibility and cost performance must be considered.\(^16,17\)
Magnesium oxide (MgO) has been investigated as one of the most efficient borate sorbents, and related compounds, such as layered double hydroxides (LDHs) based on Mg and their calcined products, have also been shown to be reusable sorbents for anionic species like borate and fluoride. Magnesium oxide is a basic material for reactive sorbents before modification, and the removal of borate and fluoride onto MgO has been previously reported. However, most investigations have focused on sorption isotherms to predict performance, and the mechanism of borate removal by MgO has not yet been fully discussed. Garcia-Soto and Camacho demonstrated three steps of reaction; (1) hydration and formation of Mg(OH)₂ gel, (2) acid-base reaction between MgO and water and subsequent diffusion of B(OH)₄⁻ into micropores, and (3) stereo-specific chemical reaction between B(OH)₄⁻ and OH⁻ by a ligand exchange model. Based on this mechanism, immobilized B species have been regarded as B(OH)₄⁻. In the present work, a mechanism for borate removal by MgO will be discussed based on observation by several techniques. Understanding the mechanism will facilitate optimization of the properties of MgO as an industrial sorbent for borate, because it is usually produced by calcination of natural magnesium (MgCO₃) and/or hydromagnesite (Mg₂(CO₃)(OH)₂).

2. Experimental

0.10–2.00 g of magnesium oxide was added to 0.04 L of the chosen concentrations (5.6–67.0 mM as B) of H₃BO₃ solution in 50 mL polyethylene bottles. The initial pH was adjusted to 9.0 ± 0.2 using 1.0 mol/L NaOH, and in some cases to 6.0 ± 0.2 and 11.0 ± 0.2. Duplicate suspensions were shaken on a rotary shaker at 100 rpm with a stroke of 7.5 cm and maintained at 25°C, until equilibrium was achieved. In some experiments, 12 mM EDTA was added as a masking reagent for Mg²⁺ ions. All chemical reagents were special grade (Wako Pure Chemical Industries, Ltd., Osaka, Japan).

At intervals, samples were removed, filtered with cellulose acetate membrane (0.2 µm pore size), and diluted for determination of the concentrations of dissolved B and Mg species by ICP-AES (VISTA-MPX, Seiko Instruments, Chiba, Japan). Samples of solid residues were lyophilized in a freeze-dryer for more than 24 h and stored in a vacuum desiccator prior to XRD, ¹¹B-NMR, SEM and FTIR.

X-ray diffraction patterns of the solid residues were collected on a RIGAKU Ultima IV XRD (Akishima, Japan) using CuKα radiation (40 kV, 40 mA) at a scanning speed of 0.02° and 2 mass% in KBr. Chemical reagents Mg(OH)₂, Na₂B₄O₇ and H₂BO₃ (special grade, Wako Pure Chemical Industries, Ltd., Osaka, Japan) were used as standards.

Fourier-transformed infrared spectra (FTIR) for the same solid residues were collected using an FTIR 670 Plus (JASCO, Hachioji, Japan) in transmission mode after dilution to 2 mass% in KBr. Chemical reagents Mg(OH)₂, Na₂B₄O₇ and H₂BO₃ (special grade, Wako Pure Chemical Industries, Ltd., Osaka, Japan) were used as standards.

SEM images of the solid residues after sorption of H₃BO₃/B(OH)₄⁻ were observed with a scanning electron microscope (SEM; VE-9800, Keyence, Osaka, Japan) using 15 kV accelerating voltage.

Results and Discussion

The sorption rate of H₃BO₃/B(OH)₄⁻ on MgO was clearly dependent on the ratio of MgO mass to solution volume, as shown in Fig. 1(a), with a greater ratio providing faster sorption. Mg²⁺ ions were released by the sorption of borate on the MgO, as shown in Fig. 1(b). It was to be expected that the maximum concentrations of released Mg²⁺ ions were negatively correlated with the MgO mass to solution volume.
ratio in the presence of the same initial [B] (Figs. 1(a), 1(b)).

The pH increased immediately after contact of MgO with the borate solution, and stronger alkalization occurred when a larger mass of MgO was used (Fig. 1(c)). The molar ratios of B/Mg in the solid residues were calculated to be 0.42, 0.22, 0.12, 0.062 and 0.053 for MgO masses of 0.10, 0.25, 0.50, 1.00 and 2.00 g, respectively.

Figure 2 shows the effect of initial B concentration on B and Mg concentration and pH in the presence of 0.10 g MgO in a 0.040 L solution. At 67.0 mM initial B concentration, equilibrium was not achieved within 120 h, as shown in Figs. 1(a) and 2(a). The maximum concentrations of released Mg$^{2+}$ ions clearly correlated with initial B concentration (Fig. 2(b)). A remarkable amount of Mg$^{2+}$ ions were released within the first 10 h, while B concentration did not change much when the initial B concentration was 67.0 mM. These results indicate that Mg$^{2+}$ ions were extracted by B species, possibly through the formation of surface complex species (≡MgO–Mg–B(OH)$_3^+$ in eq. (6)). In this case, pH increased immediately to 10.0–10.4, and less significant increases in pH were observed at higher initial B concentration (Fig. 2(c)). Molar ratios of B/Mg in the solid residues were calculated to be 0.42, 0.13, 0.08 and 0.04 for initial B concentrations of 67.0, 21.6, 11.5 and 5.6 mM, respectively.

Effect of initial pH on the sorption of H$_3$BO$_3$ on MgO is shown in Fig. 3. The isotherms were obtained using 0.10 g of MgO in 0.04 L of 1.0–67.0 mM B solution at initial pH of 6.0–11.0. At an equilibrium concentration ($C_\infty$) of less than 25 mM, the highest sorption density ($Q$) was obtained at an initial pH of 6.0. This suggests that molecular H$_3$BO$_3$ ($pK_a$ 9.23) was a trigger for reaction with the MgO surface. However, for $C_\infty$ above 25 mM, it was stereo-chemically difficult for the H$_3$BO$_3$ trimer to approach the MgO surface to form surface complexes through triboric acid, and the equilibrium pH was lower at an initial pH of 6.0 than at 9.0, leading to an insufficient increase in saturation index for Mg(OH)$_2$ and lower $Q$ values. At an initial pH of 11.0, the predominant species were B(OH)$_4^-$ and/or B$_2$O$_3$(OH)$_4^-$, which are less reactive with MgO than H$_3$BO$_3$, resulting in far smaller $Q$ values compared with those observed at initial pH of 6 and 9.

XRD patterns for solid residues recovered after the experiments of Figs. 1 and 2 are shown in Figs. 4 and 5, respectively. With the same initial B concentrations, it was to be expected that the MgO phase was more retained with increasing mass of MgO. The XRD results clearly indicated that Mg(OH)$_2$ was a secondarily formed major phase (JCPDS 44-1482). Generally, MgO is slightly soluble in water (solubility 0.0086 g/L at 30°C$^{39}$) and immediately hydrates to become Mg(OH)$_2$ with a low product of solubility ($K_{sp}$ 10$^{-10.9}$ at 25°C), as previously described.$^{30}$

$$\text{MgO} + \text{H}_2\text{O} \rightleftharpoons \text{Mg}^{2+} + 2 \text{OH}^- \quad (4)$$

$$\text{Mg}^{2+} + 2 \text{OH}^- \leftrightarrow \text{Mg(OH)}_2 \quad (5)$$

Although most diffraction peaks for Mg$_3$B$_4$O$_{12}$·7H$_2$O are overlaid by those for Mg(OH)$_2$, a peculiar peak at $2\theta = 12.7^\circ$
(d 6.86 Å) can be assigned to only Mg₇B₄O₁₃·7H₂O, which increased with increase in B/Mg molar ratio in the solid residues. Mg₇B₄O₁₃·7H₂O, as can be seen with Mg₇(OH)₇-\-B(OH)₄(B₃O₆(OH)₃), is basically in a matrix of Mg(OH)₂. With increase in B/Mg molar ratio in the solid residues, the structure of Mg(OH)₂ is transitionally changed into Mg₇(OH)₇B(OH)₄(B₃O₆(OH)₃). The d value is also close to three times of d₁₀₁ for Mg(OH)₂, possibly being attributed to long-period ordered (LPO) structures of Mg(OH)₂. More importantly, significant broadening of these and other planes was observed with increasing B/Mg molar ratio, while the (hk0) planes were not broadened but exhibited a small shift to larger diffraction angles (Fig. 6). This indicated that integration of H₃BO₃ interfered mainly in c-axis stacking of Mg(OH)₂. It is likely that a planar molecule of H₃BO₃ coordinates to Mg atoms in parallel to layer planes to disturb
regular stacking along the c-axis (Fig. 7). These phenomena were not observed in sorption of fluoride on MgO.26,27 Other phases such as Mg₂B₂O₇·7H₂O (JCPDS 19-754) and Mg₆(CO₃)₄(OH)₂·4H₂O (JCPDS 25-513) can also be seen with increasing molar ratio of B/Mg in Fig. 4, although the reliability of identification is relatively low with these index cards. In Fig. 5, a greater amount of residual MgO remained and secondary phases other than Mg(OH)₂ decreased with decreasing initial B concentration.

SEM images taken at low magnification showed that sorbent size dramatically increased after sorption of borate (Figs. A1(a)–A1(e)), and SEM at high magnification showed that their surface morphologies also changed after sorption, from granular aggregated rods to honeycomb structures (Figs. A1(f)–A1(i)). Hydration led to swelling and increase in porosity. At lower initial B concentrations the surface of the solid residues looked like rose petal debris (Figs. A1(g), A1(h)), and gradually became less defined with increasing initial B concentration (Fig. A1(j)). This suggested that the observed honeycomb morphology is characteristic to Mg(OH)₂ precipitates at ambient temperatures26,27) and that the morphologies were influenced by the lattice strain of Mg(OH)₂ bearing larger B contents.

Figure 8 shows ¹¹B-NMR spectra for solid residues recovered after sorption of 5.6–67.0 mM B on 0.10 g MgO. The molar ratio of [⁴]B/[³]B was estimated based on the ¹¹B-NMR spectra of Na₂B₄O₇ (³B × 2 and [⁴]B × 2) and H₃BO₃ ([³]B × 1) standards. It was clear that trigonal B ([³]B) was predominant over tetrahedral B ([⁴]B) in all residues, and that the molar ratio of [⁴]B/[³]B gradually increased with initial B concentration. This suggests that the molecular form of H₂BO₃ is preferentially inserted between Mg(OH)₂ layers, and that co-precipitation of B(OH)₄⁻ with Mg(OH)₂ follows after H₂BO₃ integration when excess amounts of B are present. This is a novel insight explaining the reaction mechanism of H₂BO₃/B(OH)₄⁻ with MgO. The above results were also supported by FTIR spectroscopy (Fig. A2). FTIR bands observed at around 1460–1500 cm⁻¹ were assigned to the asymmetric B–O stretching mode νₛ([³]B–O) in B(OH)₃, the band at around 1080 cm⁻¹ was assigned to the asymmetric B–O stretching mode νₛ([⁴]B–O) in B(OH)₄⁻, and the band at 978 cm⁻¹ was assigned to the stretching mode νₗ([⁴]B–O) in B(OH)₄⁻.31) The bands at around 1260–1280 cm⁻¹ were assigned to the plane bending mode δ(B–O–H) in both B(OH)₃ and B(OH)₄⁻.32) Their intensities increased with increasing initial B concentration (Figs. A2(d)–A2(g)) and decreasing MgO concentration (Figs. A2(h)–A2(k)). Signal intensities observed for tetragonal [⁴]B at around 1080 cm⁻¹ were weak and broad for solid residues recovered after sorption of 67.0 mM B on 0.1 g MgO, as shown in Fig. A2(g). There were no IR bands assignable to Mg(OH)₂ in the measured region.

Based on the ¹¹B-NMR results that trigonal [³]B was the predominant species immobilized in the solid phase, additional experiments were conducted. Figure 9(a) shows Mg²⁺ ions released from MgO reagent when 0.50 g MgO was suspended in 200 mL of 10 mM boron solution at initial pH of 6.0 and 9.0. Under these conditions, excess amounts of MgO were added in comparison with B concentration. Changes in total B concentration were not observed within 3 h after contact of MgO with the boron solutions. It was obvious that much greater amounts of Mg²⁺ ions were released at an initial pH of 6.0. pH increased with elapsed time and converged to mostly the same value after 100 min, independent of the initial pH (Fig. 9(b)), as a result of the reactions described in eqs. (4) and (5). The proportion of boron species was calculated using the measured pH values and pKₐ (Fig. 9(c)). The molecular form of boric acid, which was dominant at pH 6.0, was mainly responsible for the observed release of Mg²⁺.
Based on all of the above results, the mechanism for the immobilization of H$_3$BO$_3$/B(OH)$_4$ by MgO can be proposed as follows. Hydroxyl groups (–OH) coordinate with the surface of MgO in an aqueous system, as shown in eq. (6). Molecular H$_3$BO$_3$ preferentially reacts with the surface to form the surface complex MgO–Mg–B(OH)$_3$+, accompanied by an increase in pH, known as ligand-promoted dissolution (eq. (6)). The surface complex MgO–Mg–B(OH)$_3$+ is dissociated into MgOH and [MgB(OH)$_4$]$^+$ according to eq. (7). Equations (6) and (7) repeat until the surface is completely covered with secondary precipitates. The complex [MgB(OH)$_4$]$^+$, which has a relatively small stability constant (pK$\approx$ 1.34 ~ 1.63 at 25°C,14), precipitates as MgB(OH)$_2$ and/or Mg(OH)$_2$ ($K_{sp}$ 1.1 x 10$^{-11}$ at 25°C,33), bearing B(OH)$_3$ as shown in eqs. (8) and (9).

As mentioned above, the principal mechanism of H$_3$BO$_3$/B(OH)$_4$ immobilization is based on formation of Mg(OH)$_2$. This was further confirmed by the following results. Figure 10 demonstrates changes in B and Mg concentration with time during sorption of 1–3 mM boric acid on MgO with presence and absence of 12 mM EDTA at an initial pH of 9.0 in 1.00 L. 0.01 g MgO was added to 40 mL solution including 1–3 mM H$_3$BO$_3$ with or without 12 mM EDTA.
80 h. Its hydration, as shown in eq. (8), was blocked by EDTA as follows:

\[
[MgB(OH)4]^+ + EDTA \rightleftharpoons B(OH)4^- + [EDTA Mg]^{2+} \tag{10}
\]

The Mg\(^{2+}\) ions were stabilized in solution as a chelate complex with EDTA, which inhibited the precipitation of Mg(OH)\(_2\) and led to no immobilization of B species. Thus, it was confirmed that formation of Mg(OH)\(_2\) is principle for immobilization of H\(_3\)BO\(_3\)/B(OH)\(_4^-\).

4. Conclusions

The mechanism of H\(_3\)BO\(_3\)/B(OH)\(_4^-\) immobilization on MgO was investigated by sorption data in combination with characterization by XRD, \(^{11}\)B-NMR and FTIR of solid residues recovered after sorption. \(^{11}\)B-NMR results indicated that the predominant B species in solid residues after sorption at an initial pH of 9.0 was trigonal \(^{11}\)B. Dissolution rates of Mg\(^{2+}\) from MgO at various pH showed that the molecular form of H\(_3\)BO\(_3\) is responsible for release of Mg\(^{2+}\) from MgO. Thus, the ligand-promoted dissolution of MgO by H\(_3\)BO\(_3\) was proposed as the initiation step in the reaction of H\(_3\)BO\(_3\)/B(OH)\(_4^-\) with MgO. Through the surface complex \(\text{MgO-Mg-B(OH)4}^+\), complex ions of \([\text{MgB(OH)}_3]^+\) are released in the aqueous phase and are immediately hydrated, resulting in Mg(OH)\(_2\)-bearing H\(_3\)BO\(_3\). Formation of Mg(OH)\(_2\) is the principle mechanism of H\(_3\)BO\(_3\) immobilization, which occurs by coordination to Mg atoms and interferes in the c-axis stacking of Mg(OH)\(_2\), with production of some B(OH)\(_2\) by co-precipitation, based on \(^{11}\)B-NMR and XRD results. XRD patterns for the solid residues demonstrated more significant broadening, except forhk0 plane reflections, with increasing B content. Because these hk0 planes were not dependent on the c-axis, it was proposed that the molecular H\(_3\)BO\(_3\) between Mg(OH)\(_2\) layers led to significant structural strain along the c-axis with increasing B content of the solid residues. Effect of initial pH on the sorption density of B species (\(Q\)) depended on the equilibrium concentration of B (\(C_2\)). An initial pH of 6.0 provided a larger \(Q\), less than 25 mM C\(_e\), while an initial pH of 9.0 provided a larger \(Q\), more than 25 mM C\(_e\). This was considered to be due to the fact that at an initial pH of 6.0, the equilibrated pH did not sufficiently rise, resulting in under saturation for Mg(OH)\(_2\) to sorb B.

These findings are applicable to the sorption mechanism of H\(_3\)BO\(_3\)/B(OH)\(_4^-\) in substances related to MgO such as layered double hydroxides (LDHs) including Mg and their calcined products, and is expected to contribute to the development of novel sorbents for inorganic B species.

Acknowledgements

Financial support was provided to KS by Funding Program for Next Generation of World-Leading Researchers (“NEXT program” GR078) in Japan Society for the Promotion of Science (JSPS). This study was partially supported by the New Energy and Industrial Technology Development Organization (NEDO) under the Innovative Zero-emission Coal-fired Power Generation Project. The authors thank Dr. Kwadwo Osseo-Asare at Pennsylvania State University for discussion on dissolution of metallic oxides using JSPS Invitation Fellowship Program for Research in Japan FY2011 (Short Term ID S-11179).

REFERENCES

Appendix

Fig. A1  SEM images of MgO ((a) and (f )) and solid residues (others) recovered after sorption of 5.6-67.0 mM borate on 0.10 g MgO at an initial pH of 9.0 and 25°C at low magnification ((a)–(e)) and high magnification ((f)–(j)). Initial [B] were 5.6 mM for (b) and (g), 11.2 mM for (c) and (h), 21.6 mM for (d) and (i) and 67.0 mM for (e) and (j). Horizontal bars indicate 5.00 µm in (a)–(e) and 1.00 µm in (f)–(j).
Fig. A2  FTIR spectra for solid residues recovered after sorption of B on MgO, as shown in Figs. 1 and 2 and related standards. (a) H$_3$BO$_3$, (b) Na$_2$B$_4$O$_7$, (c) Mg(OH)$_2$, (d) 5.6 mM B with 0.10 g MgO, (e) 11.5 mM B with 0.10 g MgO, (f) 21.6 mM B with 0.10 g MgO, (g) 67.0 mM B with 0.10 g MgO, (h) 67.0 mM B with 0.25 g MgO, (i) 67.0 mM B with 0.50 g MgO, (j) 67.0 mM B with 1.00 g MgO and (k) 67.0 mM B with 2.00 g MgO.