SYNTHESIS OF Mg3(PO4)2.8H2O

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 ${\rm Mg_3(PO_4)_2}{}^{\bullet}$ 8H₂O was prepared by converting ${\rm Mg_3(PO_4)_2}{}^{\bullet}$ 22H₂O in an alkaline solution at a pH of about 9. The conversion period was reduced to 2-4 weeks by the addition of ${\rm MgHPO_4}{}^{\bullet}$ 3H₂O to the 22-hydrate and the pH adjustment with ${\rm Na_2CO_3}{}^{\bullet}$.

Although $\mathrm{Mg}_3(\mathrm{PO}_4)_2^{\bullet}$ 8H₂O (TMP8) was obtained when $\mathrm{Mg}_3(\mathrm{PO}_4)_2^{\bullet}$ 22H₂O (TMP22) was allowed to stand in an alkaline solution in about two months, ¹⁾ the conditions for the synthesis of TMP8 had not been clear in detail yet. In the present work, the preparation of TMP8 from TMP22 was investigated to reduce the time of the conversion.

TMP22 was precipitated by mixing solutions of magnesium salt and sodium phosphate²⁾ instead of the preparation by slow conversion of MgNH₄PO₄• 6H₂O (struvite) to TMP22 in water.^{1,3)} TMP22 was converted to TMP8 at 25 °C in an alkaline water at a pH of about 9 adjusted by the addition of Na₂CO₃.

MgHPO₄• 3H₂O (DMP3) was also added to TMP22 in order to reduce the conversion time. The treatment was continued until the main peaks of X-ray diffraction pattern for TMP22 disappeared. The TMP8 sample with impurities was separated from the alkaline solution by filtration. The filtered sample was treated by boiling water for 1 hr, and thus the pure sample of TMP8 was obtained.

The change of X-ray diffraction pattern with time (Fig. 1) shows that TMP22 was decomposed and TMP8 was formed in 2-4 weeks. On the other hand, it took more than 6 weeks for TMP8 to form in an alkaline water with a pH of about 9 adjusted by NaOH or in the solution from which TMP22 had been precipitated in advance.²⁾

The addition of less than 1g of DMP3 to 10g of TMP22 was ineffective for

accelerating the conversion. When more than 5g of DMP3 was added, some of DMP3 crystals unreacted were observed by microscope although neither TMP22 nor DMP3 was detected by the X-ray analysis. The most appropriate amount of DMP3 was considered to be 2-3g to log of TMP22; all the crystals of TMP22 and DMP3 disappeared at the final stage of the conversion.

The pattern of the sample treated by boiling water (Fig. 2) was identical with that of ASTM X-ray Powder Data File 16-330. The comparison of the X-ray patterns of the samples before and after the boiling treatment indicates that unknown contaminates in the TMP8 sample might be dissolved in the boiling water.

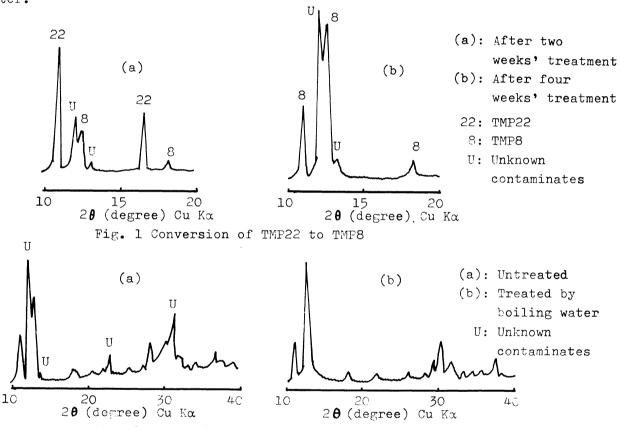


Fig. 2 X-ray diffraction patterns of TMF8

References

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