Occurrence of magnetic spherules in the Maastrichtian bone bed sedimentary sequence of Fatehgarh Formation, Barmer basin, India

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We report the presence of magnetic, glassy spherules of millimetre size along with fine magnetic particles in the sedimentary sequence of Fatehgarh Formation of Barmer Basin, associated with Maastrichtian mass extinction fossils. In the present study, XRD, XRF, EDXA and Mössbauer spectroscopy are employed for the preliminary characterization of material recovered from this horizon. Studies indicate predominant presence of iron and titanium in spherules, while the sedimentary host rock in which the spherules occur contains rare earth elements but is devoid of titanium. The presence of spherules indicates that some high-temperature event has occurred in the Maastrichtian era, which was hitherto unknown.

Keywords: Bone bed, Fatehgarh Formation, impact and volcanic activity, magnetic spherules, Maastrichtian.

FATEHGARH Formation in Barmer district of Rajasthan encompasses Cretaceous sedimentary sequence, including a strata rich in bone beds of Maastrichtian age[1]. We report here the occurrence of magnetic spherules within the sedimentary rock associated with this bone bed. Some of the spherules are glassy, which resulted due to quick quenching indicative of their high-temperature origin, and show fractures due to severe stress they faced after solidification. These spherules show narrow size distribution ranging between 0.1 and 1.5 mm.

Spherules are formed in several ways in nature[2], e.g. by volcanism, as ablation products of meteorites as they pass through the earth's atmosphere, melting of interplanetary dust particles (IDP) in the upper atmosphere as they get heated by atmospheric friction and by large impacts of asteroids and comets, for example, as it happened at the Cretaceous–Tertiary boundary, KTB. The possible geological, palaeontological, biological, chemical and physical effects of the meteoritic impact at KTB can be found in several publications[3–6].

Spherules are originally formed in a wider size range by natural processes such as volcanism, meteorite ablation or by large impacts. However, the narrow size distribution of the spherules observed in the present investigation indicates that they have been sorted by transportation or some other process before deposition in Barmer sediments. The possibility of spherules originally being formed by melting of IDP can be ruled out as they normally give rise to spherules in a narrow size range (microns to millimetres)[7]. Moreover, IDP contain large concentration of nickel, but as discussed later, spherules do not contain Ni.

To understand the association of these spherules with the Barmer basin, we briefly describe some features of the Barmer basin[8–10].

Barmer basin was formed when the Indian craton broke up, towards the end of Cretaceous and led to the formation of the Cambay rift and constituent basins. Barmer basin is one of the constituent basins. It is a narrow north-south trending graben that comprises sediments of Middle Jurassic to Lower Eocene age[8–10]. The sediments of Cretaceous age of Barmer basin are named as Fatehgarh Formation and are best exposed at Lordi Nala in the vicinity of Fatehgarh (26°26.087′N, 071°12.519′E). In the Fatehgarh Formation, there exists a few centimetres thick bed, hereafter named as bone bed, which is rich with fossils of microvertebrates of Late Maastrichtian age, such as *Igdabetis* species along with *Semionodontid*, *Lepisosteus indicus*, *Enchodontidae indet*, *Labiridae indet*, etc.[1,11]. This bone bed layer has been found to extend for at least about 15 km and contains fossils of a large variety of species. This is considered to be an evidence of mass mortality. The simultaneous presence of spherules and index fossils of *Igdabetis* species in the same horizon indicates that the spherules were formed in the Late Maastrichtian[11]. It should be noted that the KTB event also occurred at the end of the Maastrichtian.

To understand the origin of these spherules, we have carried out their petrography and chemical analyses by X-ray diffraction (XRD), X-ray fluorescence (XRF), energy dispersive X-ray analysis (EDXA) and Mössbauer spectroscopy. The results are described here. Figure 1 *a* and *b* shows magnetic spherules and magnetic dust. The petrographic investigation shows that many spherules have a well-defined rim and indicates the presence of glassy material. Some typical photomicrographs are shown in Figure 2.

In the present investigation we have collected sediment samples, i.e. host rock (samples A and B) from two different sites of the bone bed separated by a distance of about 10 km. A hand magnet was used to separate the magnetic portion from these samples, which include spherules as well as fine dust, both being highly magnetic. Only a few grams of spherules and a few hundred grams of magnetic dust were obtained from hundreds of kilograms of samples A and B.

Figure 3 shows the XRD pattern of powder made from spherules using CuK_α line. The petrographic studies indi-

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a *b*

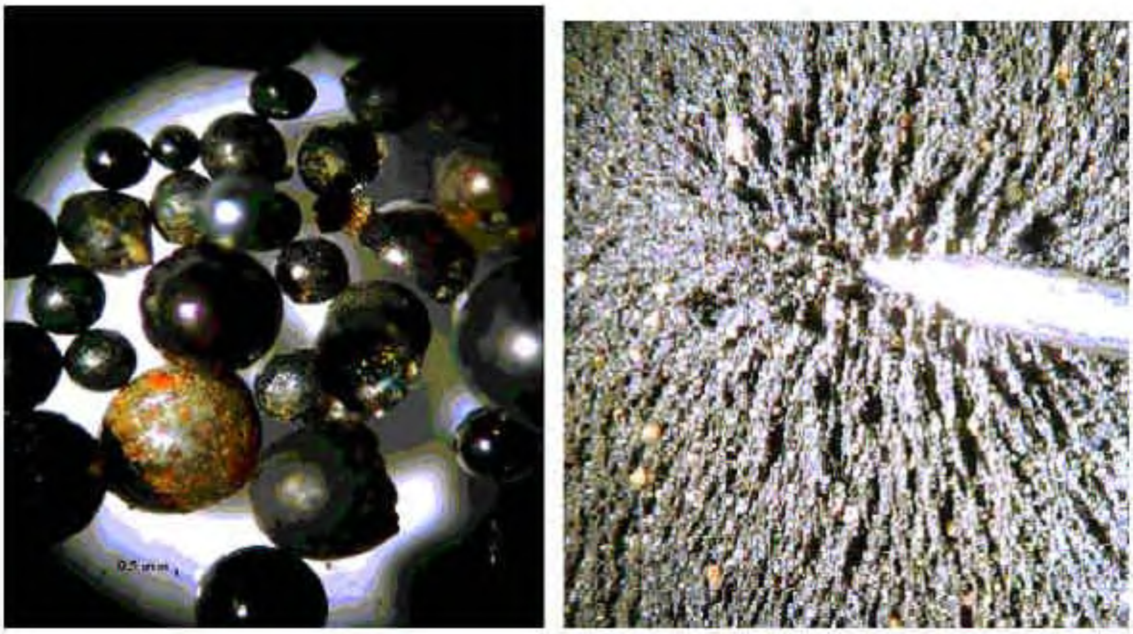

Figure 1. Optical micrographs of (***a***) spherules found in the top layer of the bone bed and (***b***) magnetic dust recovered from the bone bed.

Table 1. Elemental concentration and other details of spherules using EDXA

	Spherules			
	1	2	3	4
Element	Weight%	Weight%	Weight%	Weight%
O	37.73 ± 6.65	44.95 ± 8.52	43.01 ± 0.01	53.75 ± 0.02
Fe	40.71 ± 7.13	36.56 ± 7.52	47.28 ± 0.01	13.48 ± 0.01
C	14.40 ± 11.26	10.94 ± 12.95		
Al	2.48 ± 1.17		4.45 ± 0.01	3.01 ± 0.00
Si	2.37 ± 1.18	1.49 ± 1.05		4.10 ± 0.00
Ca	1.38 ± 0.98	2.20 ± 1.29	2.23 ± 0.01	2.26±0.00
Ti		2.25 ± 1.59	0.64 ± 0.00	15.49 ± 0.01
Cl			1.28 ± 0.01	
Mn				6.84 ± 0.01

cate the presence of glassy material and Mössbauer spectroscopic studies (discussed later) show the presence of nano-size particles. Hence the broad humps on large background of XRD pattern of spherules can be attributed to a mixture of nano-crystalline and glassy material. In Figure 4 *a* we display the XRD pattern of magnetic dust. This pattern also shows similar peaks, indicating presence of nano-crystalline and glassy material in magnetic dust. In Figure 4 *b* and *c* we display the XRD pattern of sedimentary host rock samples A and B, which indicates that the host rock basically consists of crystalline phases together with minor glassy component. The presence of glassy phase in spherules and magnetic dust indicates that they were formed in some high temperature process accompanied by fast quenching.

EDXA studies were carried out on a representative sample of spherules, magnetic dust and host rock. We have randomly selected four spherules for EDXA studies. The EDXA spectrum of each spherule was obtained after breaking it into two parts. Table 1 shows the concentration of various elements observed from EDXA. It can be seen that spherules contain a large concentration of iron. Spherules also contain appreciable amount, of other elements like Ti, Al, Si and C. The EDXA data obtained for the two fractions of magnetic dust are also given in Table 2. These fractions again show dominating presence of iron and considerable amounts of other elements like Ti, Al and Si.

Table 3 shows the concentration of various elements present in the fraction of host rocks samples A and B. It can be seen that iron is present as the dominating phase

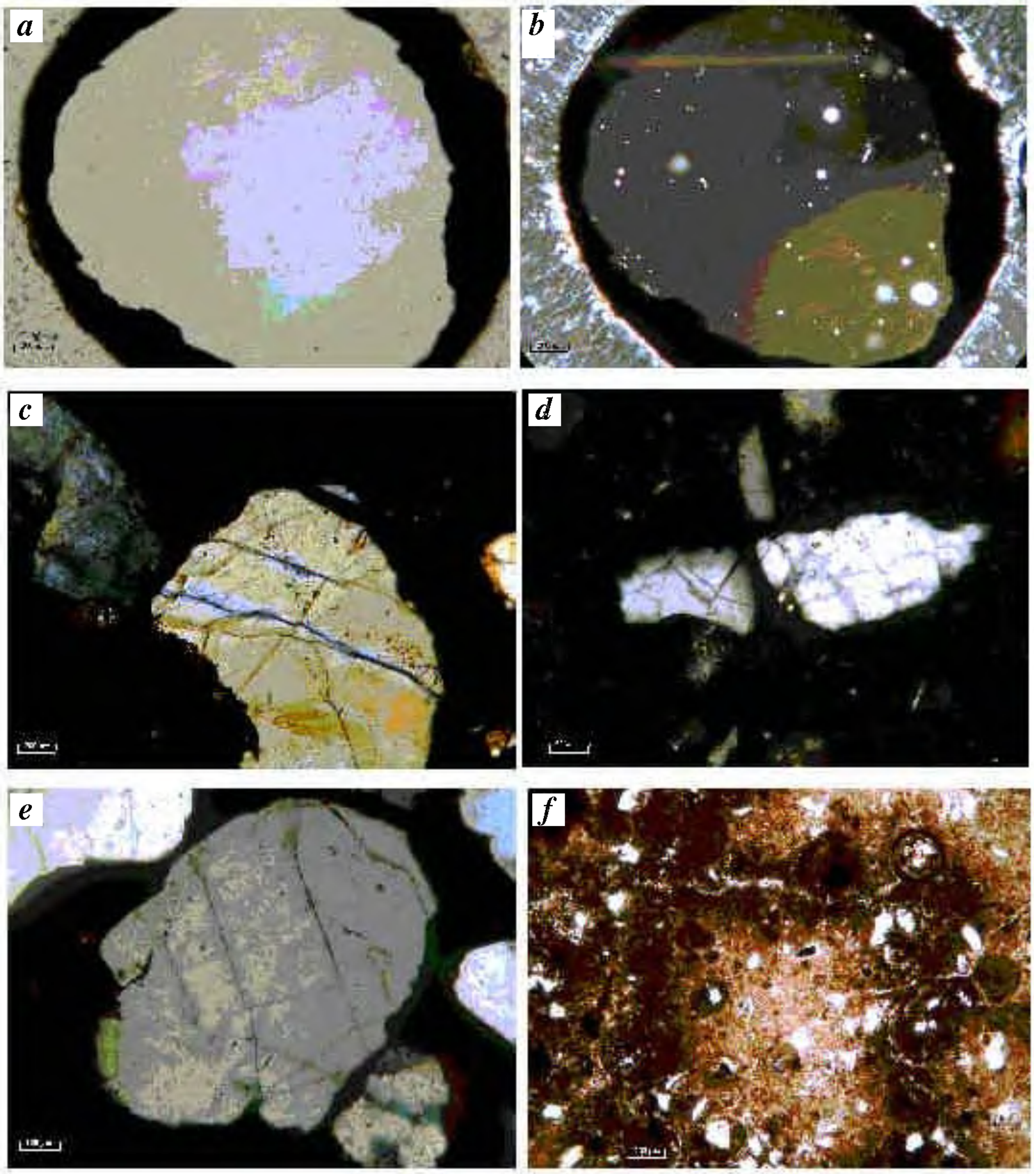

Figure 2. ***a***, Photomicrograph of a spherule section in polarized light showing glass grain enveloped by a thin opaque rim. ***b***, Same spherule in cross-polarized light. The glass grain is isotropic (the dots are contamination on the surface of the slide). ***c***, Photomicrograph of a quartz grain enclosed in spherule showing parallel fractures that must have developed due to shock (polarized light). ***d*** and ***e***, Magnified view of deformed quartz grains in associated sandstone. Quartz grains show irregular extinction and sets of parallel fractures, possibly PDFs (cross-polarized light). ***f***, Heterogeneous mixture of spherules, glass, small rock and mineral fragments. Flow banding is clearly visible in the glass (polarized light).

Table 2. Elemental concentration and other details of magnetic dust using EDXA

	Magnetic dust	
	1	2
Element	Weight%	Weight%
O	46.62 ± 2.29	47.04 ± 2.63
Fe	39.08 ± 2.52	35.81 ± 2.78
Al	4.23 ± 0.85	5.08 ± 0.94
Si	6.90 ± 0.98	7.94 ± 1.09
Ti	3.17 ± 0.86	1.22 ± 0.71
Mg		1.87 ± 0.85

together with elements like Si, Ca, Al, Mn and P. Other elements may be present below detection limit of the instrument.

We have carried out XRF studies on magnetic dust and host rock samples only, since the amount of spherules available was not sufficient for this study. In this system, 100 mCi Am source was used with the absorber holder having 16 mm diameter. The XRF spectra for magnetic dust, and host rock samples A and B are shown in Figure 5 *a–c* respectively. Details of assignment of various peaks and XRF data are shown in Tables 4–6. It can be inferred

Table 3. Elemental concentration and other details of host rock using EDXA

	Host rock sample A		Host rock sample B	
	Part-1	Part-2	Part-1	Part-2
Element	Weight%		Weight%	
O		60.71 ± 0.05	51.20 ± 2.68	52.15 ± 0.05
Fe		10.54 ± 0.02	24.05 ± 2.59	22.15 ± 0.02
C	33.90 ± 5.72			
Al	61.20 ± 5.34		3.32 ± 0.81	1.96 ± 0.00
Si		14.38 ± 0.02	12.43 ± 1.29	7.20 ± 0.01
Ca		9.50 ± 0.01	4.05 ± 0.84	10.20 ± 0.01
Mn			2.27 ± 1.26	1.08 ± 0.01
P		2.16 ± 0.03	1.66 ± 0.69	1.68 ± 0.03
Ir	4.84 ± 1.17			

Table 4. XRF data and assignment for magnetic dust

Peak energy (keV)	Elements assigned
6.40	Fe
7.06	Fe
10.55	Pb
12.60	Pb
14.76	Pb
34.70	Ce
48.52	Gd

Table 5. XRF data and assignment for host rock sample A

Peak energy (keV)	Elements assigned
2.79	Tc
3.73	Te
6.41	Fe
7.06	Fe
9.17	Ir
10.55	Pb
12.61	Pb
15.75	Zr
18.74	Nb
20.45	Tc
26.29	Sb
27.42	Tc
28.54	Sn/I
30.97	Cs
32.19	Ba
32.96	I/La
33.42	La
34.20	Ce
34.69	Ce
35.47	Pr
35.98	Cs
36.78	Nd
37.32	Nd
39.19	Ce
40.10	Sm
42.21	Nd
48.45	Eu/Gd

Table 6. XRF data and assignment for host rock sample B

Peak energy (keV)	Elements assigned
6.42	Fe
7.06	Fe
10.55	Pb
12.63	Pb
14.14	Sr
14.90	Y
15.83	Sr
24.83	Ag
25.22	Sn
27.40	Te
31.74	Te
32.17	Ba
32.96	I/La
33.41	La
34.21	Ce
34.70	Ce
35.46	Pr
35.99	Cs/Pr
36.77	Nd
37.33	Nd
39.19	Ce
40.10	Sm
40.74	Pr
42.20	Nd
42.95	Gd
45.18	Pm/Dy
48.43	Eu/Gd

that iron is present as the dominating element in all the samples, as also observed in EDXA. In XRF, we have also found evidence for appreciable presence of rare earth elements (REE) in host rock samples.

On the basis of EDXA and XRF studies we can draw the following conclusions: (i) Iron is present in appreciable amounts in all the samples. (ii) Spherules have appreciable amounts of Ti. (iii) Host rock is rich in REE but devoid of Ti, indicating that spherules were externally incorporated in sediments. (iv) magnetic dust is devoid of REE.

^{57}Fe Mössbauer spectroscopy serves as a useful technique for characterizing the chemical state of iron in geo-

Table 7. Mössbauer parameters of spherules

IS (mms^{-1})	QS (mms^{-1})	LW (mms^{-1})	HMF (koe)	Area (%)
Spherules at room temperature				
0.3095	0.6752	0.4236		14.5
0.8162	1.0374	1.0743		24.2
0.2515	–0.0075	0.3949	491.9 (sextet-1)	14.0
0.5863	–0.0749	0.9680	456.9 (sextet-2)	20.0
0.1956	–0.1748	1.0224	324.8	15.6
0.0580	–0.1077	0.8503	241.7	11.7
Spherules at low temperature (120 K)				
0.4167	0.4656	0.3514		3.5
0.0962	1.4014	0.5186		4.4
0.8128	1.7487	0.6094		8.4
0.3694	0.0132	0.5033	509.3	13.1
0.4909	–0.1384	1.3191	461.2	51.8
0.0117	–1.1958	1.3806	282.8	7.2
0.3675	0.1747	0.8963	264.7	11.5

Table 8. Mössbauer parameters of magnetic dust

IS (mms^{-1})	QS (mms^{-1})	LW (mms^{-1})	HMF (koe)	Area (%)
Magnetic dust-1 at room temperature				
0.3391	–0.1013	0.5682	510.6	59.2
0.6716	0.0245	0.5357	461.8	40.7
Magnetic dust-2 at room temperature				
0.35	–0.061	0.88	507.3	57.4
0.68	0.088	0.37	460.2	22.9
0.89	0.29	0.64	400.7	12.7
0.54		0.79		5.5
–0.018		0.34		1.5

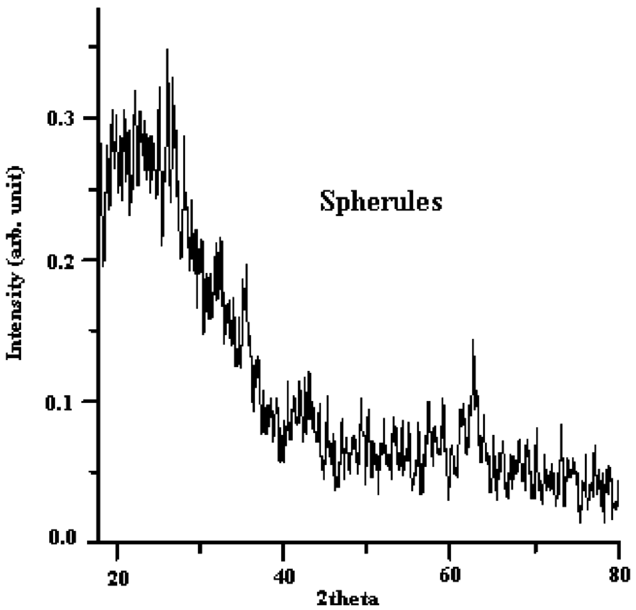

Figure 3. X-ray diffraction pattern of magnetic portion of crushed spherules separated by hand magnet.

Table 9. Mössbauer parameters of host rock sample A

IS (mms^{-1})	QS (mms^{-1})	LW (mms^{-1})	HMF (koe)	Area (%)
At room temperature				
0.3837	0.9818	0.7413		4.6
0.4344	1.5743	0.2378		3.5
0.0902	–0.1778	0.5735	334.0	20.2
0.5421	–0.1082	0.7295	333.9	32.2
–0.0228	–0.2676	0.8335	236.7	24.5
0.8238	0.1455	0.6566	232.1	15.0
At low temperature (120 K)				
0.5055	2.2105	0.1998		2.3
0.5580	–0.0663	0.3092	457.5	19.7
0.3383	–0.1409	0.3756	457.7	34.3
0.4232	–0.1099	0.8125	419.3	43.7

Table 10. Mössbauer parameters of host rock sample B at room temperature

IS (mms^{-1})	QS (mms^{-1})	LW (mms^{-1})	HMF (koe)	Area (%)
0.3726	–0.2600	0.5549	370.9	60.9
0.4700	–0.1527	0.8328	318.7	39.1

logical samples. Several workers have used this method to characterize volcanic material, e.g. volcanic glasses[12]. On the basis of Mössbauer study, the distribution pattern of iron in volcanic glassy material (obsidian) is now well understood. It was found that irrespective of the nature of volcanism, acidic or basic, the volcanic glassy material always exhibits a characteristic Mössbauer spectrum[13,14], with intense doublet/doublets with isomer shift (IS) value centred around 1.1 mms^{-1} and quadrupole splitting (QS) value more than 2.0 mms^{-1}. Apart from glassy material, in case of intense volcanic activity, we also expect deposition of lava minerals like olivine or pyroxene or their weathered

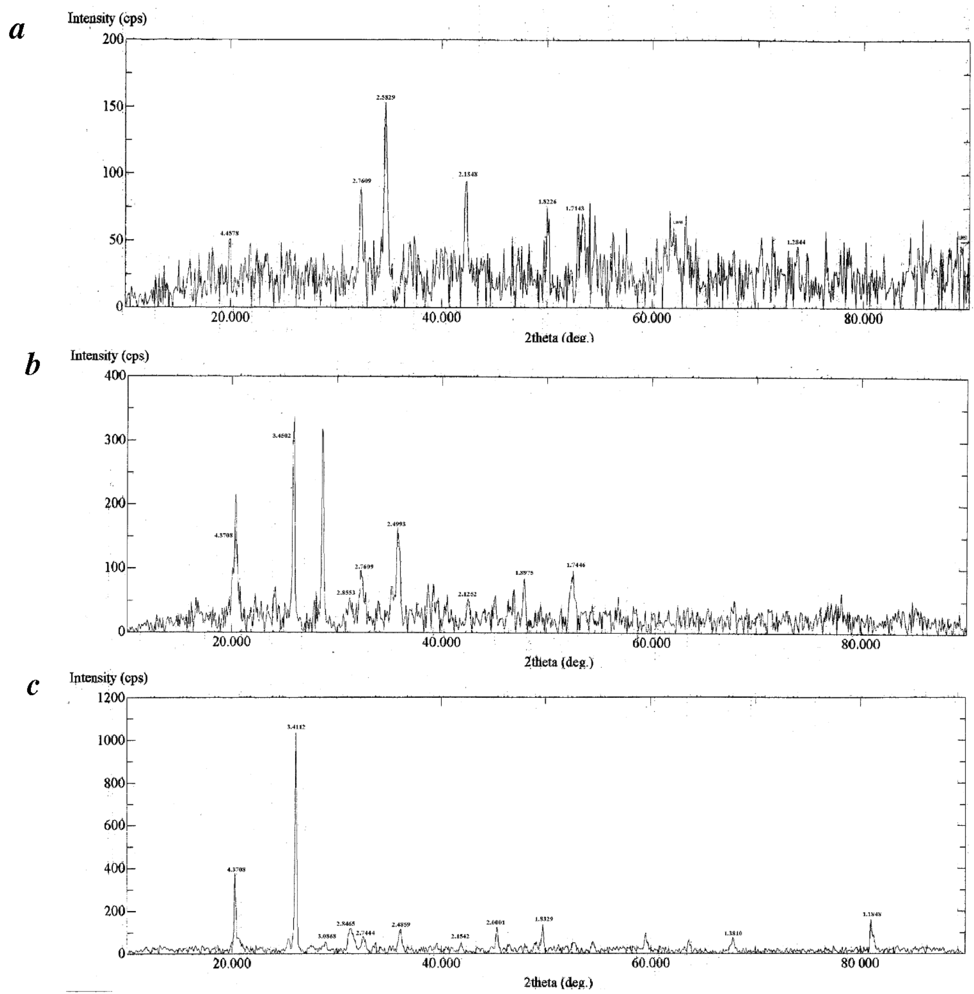

Figure 4. X-ray diffraction pattern of (***a***) magnetic dust, (***b***) powder made from host rock sample A and (***c***) powder made from host rock sample B.

products in which iron is present in some clay-forming minerals and/or hematite[15]. We also expect the presence of various sulphur-containing minerals like pyrite or jarosite, which are present in volcanic ash. These minerals always show a characteristic Mössbauer pattern and can be identified in Mössbauer spectra of sedimentary samples. It is therefore worthwhile to note the presence or absence of these volcanic minerals or minerals formed in igneous activity. Details of Mössbauer parameters of the minerals expected in basaltic rocks and sediments are available in the literature[12,16,17].

To distinguish between volcanic and impact origin of spherules, we have carried out Mössbauer spectroscopic study of spherules and related material to find any evidence of material of volcanic origin.

For this purpose, several spherules were ground into fine powder and their spectra were recorded at room temperature (RT) and at 120 K. The spectra are displayed in Figure 6 *a* and *b* respectively, and Mössbauer parameters in Table 7. The Mössbauer parameter are IS, QS, HMF (splitting due to magnetic hyperfine splitting) and LW (line width). IS is reported w.r.t. the centroid of standard Fe.

It can be seen from Table 7 that Mössbauer parameters of doublets obtained for spherules show IS and QS values characteristic of iron in Fe^{3+} state. This is typically different from volcanic glassy material, where iron is dominantly present in Fe^{2+} state. The absence of Fe^{2+} doublet in spherules is one of the important observations which may exclude volcanic origin. For the sake of comparison we give Mössbauer spectrum of volcanic glass (collected by H. C. Verma, IIT, Kanpur) in Figure 6 *c*. One can see that the spectra of spherules and volcanic glass are characteristically different.

Room temperature Mössbauer parameters of the outer two sextets (i.e. sextets 1 and 2) of spherules are close to that of magnetite or titanomagnetite. If we attribute these sextets to iron in A and B sites of magnetite or titanomagnetite, then at lower temperature (120 K) we should get a large increase in magnetic hyperfine splitting for both A and B sites; but we have not noticed such increase

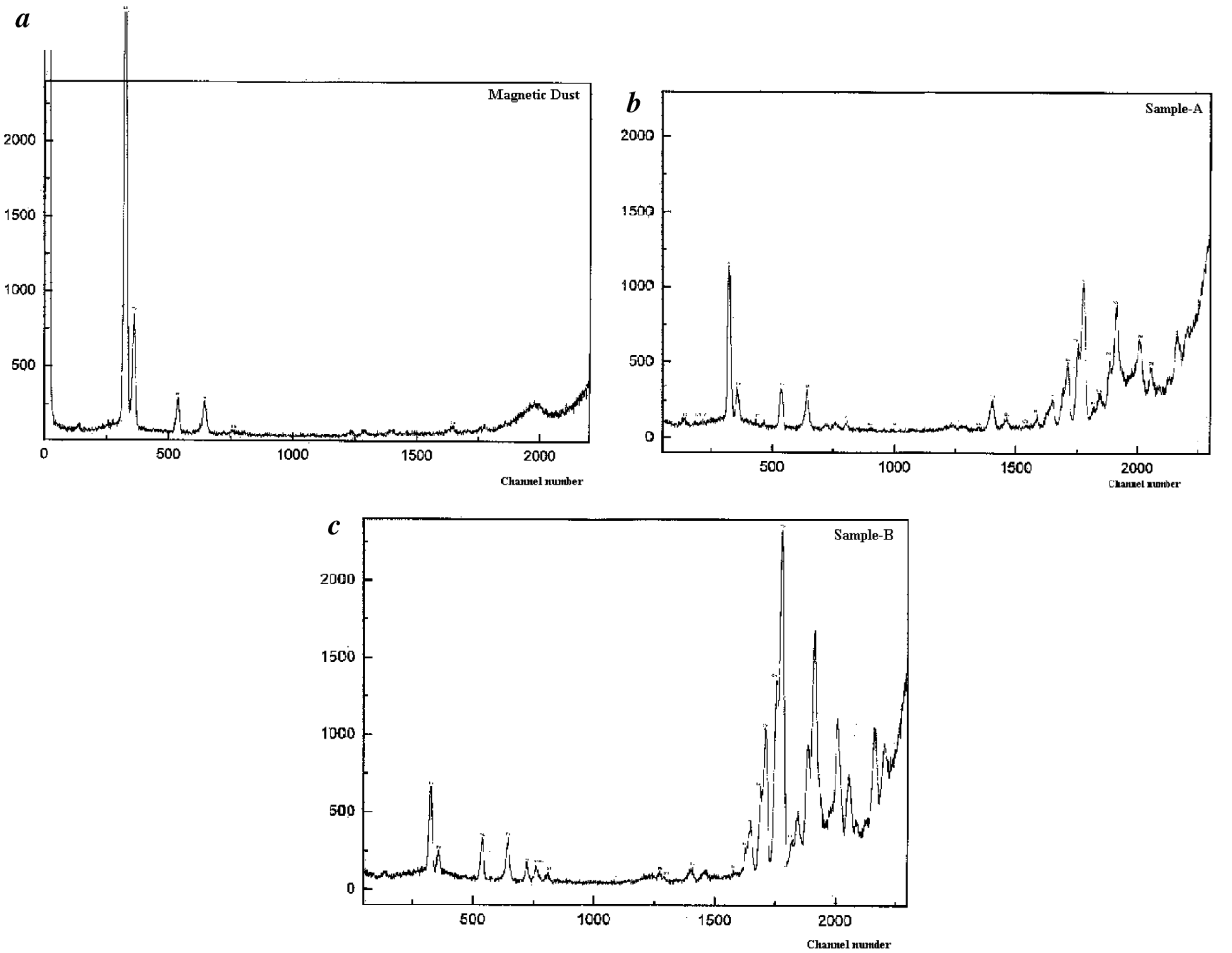

Figure 5. X-ray fluorescence yield of (***a***) magnetic dust, (***b***) host rock sample A and (***c***) host rock sample B. (*Lead peaks are for calibration purpose.)

(c.f. Table 7). We therefore infer that these sextets are not due to pure magnetite and may belong to some complex spinel phase.

The Mössbauer spectra of two fractions of magnetic dust are displayed in Figure 7 *a* and *b* respectively, and Mössbauer parameters are given in Table 8. Both fractions show the presence of two sextets (labelled 1 and 2), whose Mössbauer parameters are close to those obtained for the outer two sextets of spherules. Thus, it appears that iron is present in some complex spinel phases in both spherules and magnetic dust.

The Mössbauer spectra of host rock sample A as recorded at RT and 120 K are shown in Figure 8 *a* and *b* respectively. Mössbauer spectrum of host rock sample B recorded at RT is shown in Figure 8 *c*. Mössbauer parameters for host rock samples A and B are given in Tables 9 and 10 respectively.

In case of host rock sample A, we obtained a relaxed spectrum (Figure 8 *a*), indicating the presence of iron in nano phase[18]. When we cool this sample to 120 K (Figure 8 *b*), we get a full-grown sextet characteristic of goethite (Table 9). Similarly, Mössbauer spectra of sample B collected at RT (Figure 8 *c* and Table 10) exhibit the presence of two sextets with negative QS. Mössbauer parameters of the outer sextet are characteristic of goethite. It appears that in host rocks iron is mainly present as goethite and shows no signs of any volcanic mineral.

Thus based on Mössbauer spectroscopy we have not found evidence of any iron-containing mineral, which is typical of volcanic activity.

The association of spherules with the Maastrichtian vertebrate fossils in the sedimentary rocks is the main finding of our study. The evidence of magnetic and glassy nature of spherules indicates a high-temperature event followed by quick quenching. Our studies also rule out the formation of spherules from IDP, as well as their origin as ablation products of meteorites. Since the end Maastrichtian is associated with *K/T* boundary mass extinction,

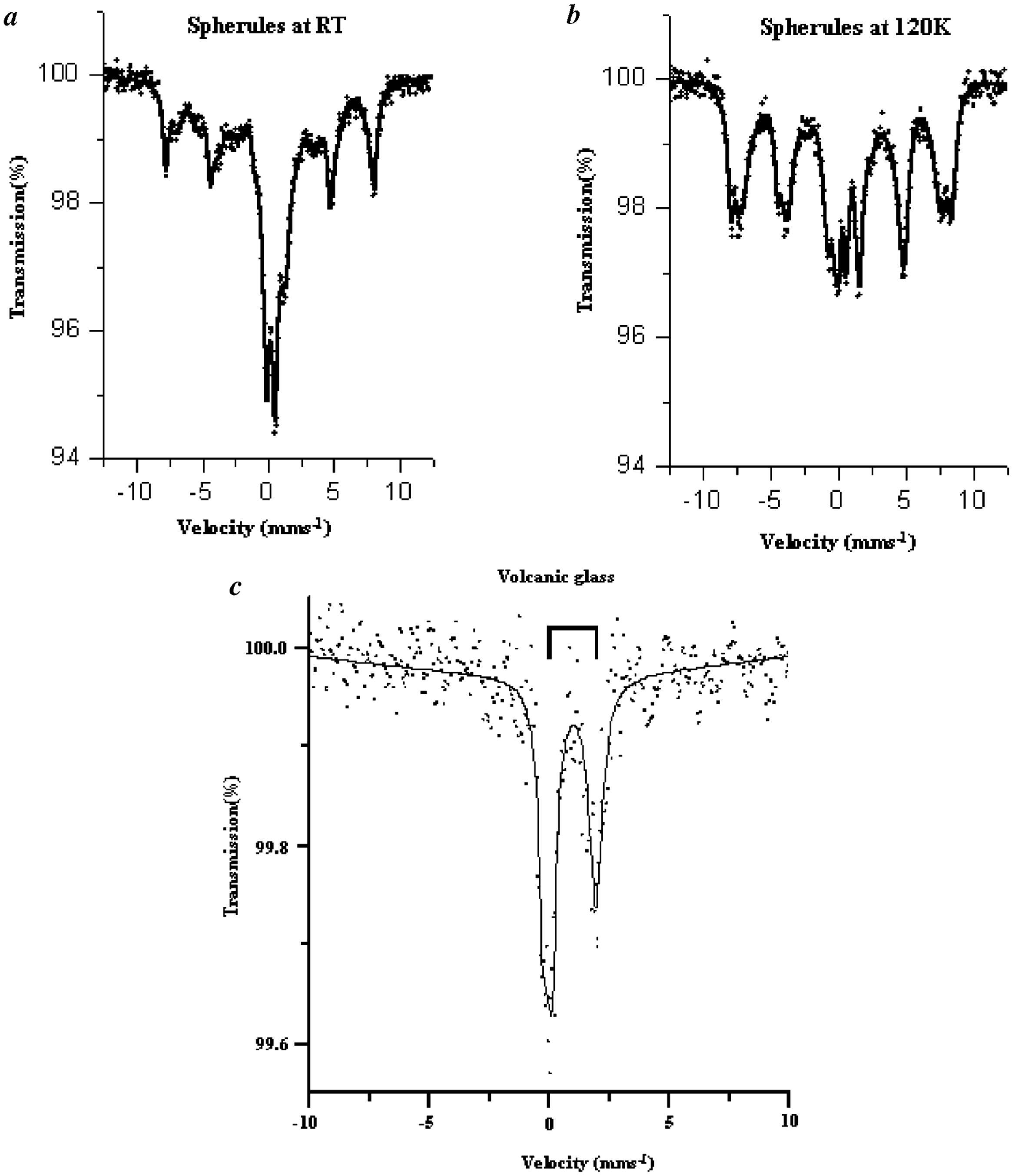

Figure 6. Mössbauer spectra of powdered spherules at (***a***) room temperature and (***b***) low temperature (120 K), and (***c***) of volcanic originated glass at room temperature.

it is important to establish if the spherules have impact origin. On the other hand, the site is close to the Deccan volcanic province and therefore, it is also essential to explore the possibility of their volcanic origin. Although presence of Ti and Fe in spherules favours volcanic origin, the absence of any volcanic mineral and lack of iron in Fe^{2+} state in spherules, as revealed by Mössbauer spectroscopic studies is against volcanic origin.

Further studies are needed to distinguish these two alternative origins of spherules. KTB clay shows the presence of platinum group elements[19], and therefore presence of Ir, Os and other platinum group elements can be the decisive evidence of KTB-related impact origin. However, it should be noted that there exist some asteroids, e.g. Vesta represented by achondritic eucrites which are poor in Ni and platinum group elements, and consistent with the

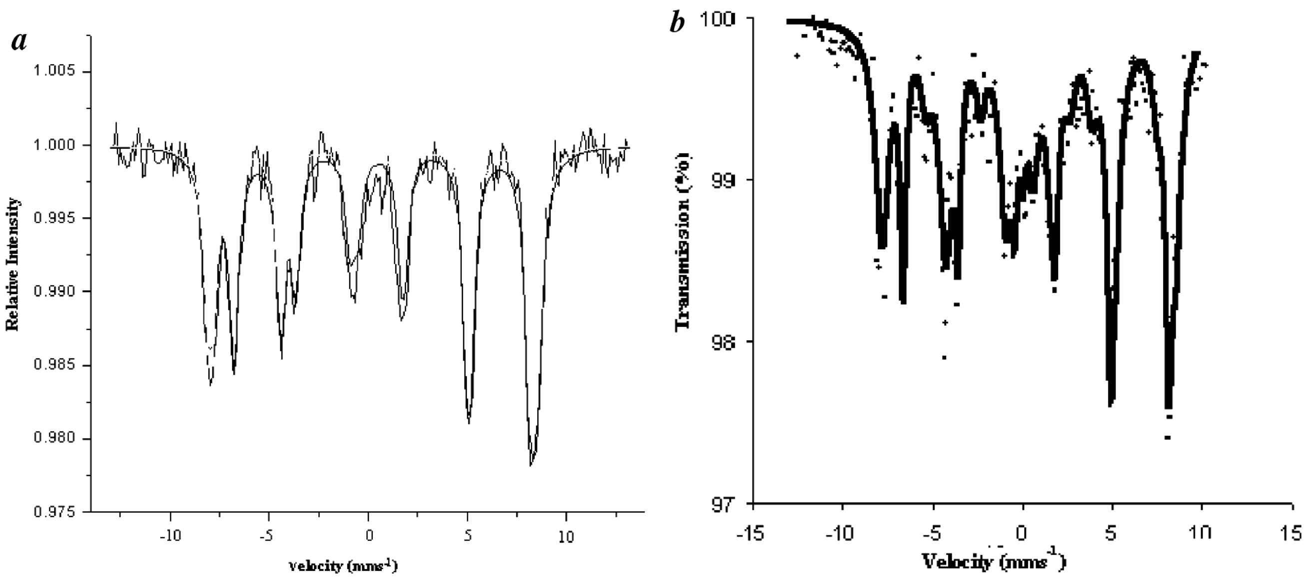

Figure 7. Mössbauer spectra of (***a***) magnetic dust-1 and (***b***) magnetic dust-2 at room temperature.

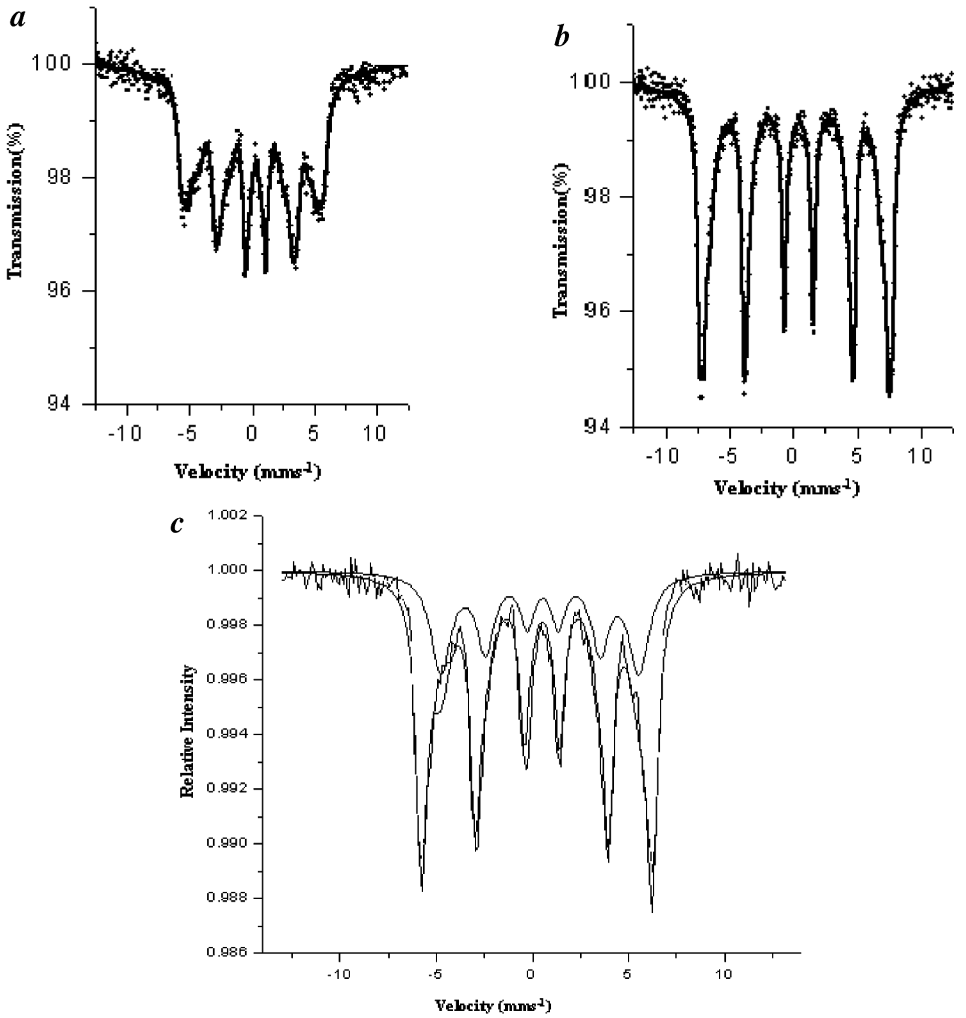

Figure 8. Mössbauer spectra of host rock sample A at (***a***) room temperature and (***b***) low temperature (120 K), and (***c***) host rock sample B at room temperature.

analysis of spherules described above. On the other hand, moon rocks are rich in Ti. Detailed work to establish the chemical nature of the material recovered from Barmer basin is now in progress.

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ACKNOWLEDGEMENTS. S.C.M. acknowledge DST, New Delhi for financial support. A.T. acknowledges CSIR, New Delhi for Research Associateship. We are grateful to Prof. H. C. Verma, IIT Kanpur for Mössbauer analysis and Dr B. L. Ahuja, Mohan Lal Sukhadia University, Udaipur for support in XRF and XRD studies. R.P.T. and A.T. are grateful to Dr J. K. Sharma, Department of Physics and M.S. Sisodia, B. S. Paliwal, Department of Geology, JNV University, Jodhpur, for help during this work. We also acknowledge the reviewer for valuable suggestions.

Received 12 April 2005; revised accepted 11 July 2005

Biosafety concerns on the use of *Photorhabdus luminescens* as biopesticide: experimental evidence of mortality in egg parasitoid *Trichogramma* spp.

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***Photorhabdus luminescens*, a symbiotic bacterium associated with the entomopathogenic nematode *Heterorhabditis indica*, has recently been shown to exhibit biopesticidal potential against important pests, independent of its host nematode. The bio-ecological compatibility of the bacterium was tested *in vitro*, against two species of the common biocontrol agent *Trichogramma* in their hyperparasitized form inside the host eggs of the rice grain moth, *Corcyra cephalonica*. In 65% of the eggs exposed to *P. luminescens* cells alone or their toxins, the *Corcyra* egg-shells became flaccid and there was significant reduction of up to 84% in the emergence of *Trichogramma* adults. The possible access of bacterial cells or their secreted toxins to the *Trichogramma* embryo sheltered inside the *Corcyra* eggshell is discussed. The nematode *H. indica* carrying the bacterium within its gut had no effect on the emergence. The results point to the bio-ecological hazards of indiscriminate use of *P. luminescens* as a biopesticide. Due to its wide host range, the inclusion of *P. luminescens* in any integrated pest management programme would be suspect, until proven safe for natural enemies and non-target organisms.**

Keywords: Biopesticide, biosafety, *Heterorhabditis indica*, *Photorhabdus luminescens*, *Trichogramma*.

THE motile Gram-negative bacterium *Photorhabdus luminescens* Thomas & Poinar (Enterobacteriaceae) found in symbiotic association with the entomopathogenic nematode

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