Tracing the maceral origin in combustion chars. The inertinite in Combustion WG of ICCP.

Report of the 1998-99 Round Robin microscopy exercise on medium rank inertinite-rich coal chars

ICCP

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REPORT OF THE INERTINITE IN COMBUSTION 1999 ROUND ROBIN EXERCISE

by Angeles Gómez Borrego

Introduction

This report is a compilation of the results of microscopy analysis on coal chars carried out during the year 1998 and 1999. The report consists on two sets of analysis performed in successive years on BB2 coal char (labelled as coal N in the CD of maceral identification in coal char). The difference between the two sets of results is that discussion occurred at the 1998 meeting on selected images. In 1999 an additional set of results come from Collinsville coal char (labelled as coal L in the CD of maceral identification in coal char). This coal char was part of the Combustion WG round robin exercise and was also used to test the inertinite in combustion WG classification system in a medium volatile bituminous coal. In addition, at the meeting in Porto it was decided to arrange a CD exercise in which participants would be asked to classify a set of images. The results of the CD exercise are part of an independent report.

Classification system of carbonaceous material in chars

The classification system used was the same one as in 1997, which essentially distinguishes between vitrinite and inertinite-derived materials, and, within the latter, porous *vs.* dense, isotropic *vs.* anisotropic and fused *vs.* unfused. This system is addressed at distinguishing those materials which differ in aspects which are thought to be of the major relevance during combustion: plastic behaviour, optical texture and porosity development.

Group 1 would include all kind of domains which could derive from vitrinite, and these can be isotropic or anisotropic, depending on the rank of the coal.

Group 2 would include the char materials which have been presumably formed from inertinites, but have been highly altered during pyrolysis, showing both anisotropic texture and an important porosity development (ρ > 50%).

Group 3 would include those domains formed from inertinites which have developed an appreciable anisotropy without further porosity development (ρ < 50%).

Conversely, the **Group 4** materials would be those which have passed though a plastic stage and generated a porous structure (ρ > 50%) but do not show any signs of anisotropy.

Group 5 would include the isotropic dense domains with evidence of having fused (i.e.: small spherical degassing pores) (ρ < 50%).

Group 6 would include all the least altered inertinites not showing cellular structure. They will be mainly massive isotropic material without any sign of having passed through a plastic stage.

Group 7 would only include the unchanged fusinites. They will be typically isotropic but might also exhibit wavy-like anisotropy.

<table>
<tr><td colspan="3">VITRINITE</td><td colspan="2">GROUP 1</td></tr>
<tr><td colspan="3"></td><td>POROUS</td><td>DENSE</td></tr>
<tr><td rowspan="4">INERTINITE</td><td rowspan="2">FUSED</td><td>ANISO</td><td>GROUP 2</td><td>GROUP 3</td></tr>
<tr><td>ISO</td><td>GROUP 4</td><td>GROUP 5</td></tr>
<tr><td rowspan="2">UNFUSED</td><td>MASSIVE</td><td colspan="2">GROUP 6</td></tr>
<tr><td>FUSINOID</td><td colspan="2">GROUP 7</td></tr>
</table>

Characteristics of the parent coals and conditions for char preparation

Some characteristics of the parent coals from which chars were prepared are shown in Table 1. Coal BB2 (N) is a virtually vitrinite-free coal with relatively low volatile matter yield for the vitrinite reflectance of this coal (Rr=0.66%). Collinsville is a coal with similar amount of vitrinite and inertinite and a medium volatile matter content. Both coals generate vitrinite-derived particles with very different characteristics.

Table 1. Petrographic analyses, volatile matter yield and classification of the parent coals

	Coal	Rr (%)	V (%)	I (%)	L (%)	VM-daf (%)	ISO	ASTM	Country
BB2	N	0.66	1.6	97.2	1.2	24.8	MRC	hvb	AU
Collinsville	L	1.28	54.2	45.8		22.3	MRB	mvb	AU

Rr=vitrinite random reflectance; V=vitrinite, I=inertinite; L=liptinite; VM=volatile matter; daf=dry-ash-free basis.

The chars were obtained at 1300 ºC from the coals ground and sized to 36-75 µm in a Drop Tube Reactor.

Instructions given for the manual analysis:

Participants were asked to classify point occurrences, in much the same way as in a coal maceral analysis and, consequently only carbonaceous material (**NOT PORES or Mineral matter**) will be counted. When the crosswire lands on carbonaceous material, a decision between any of the seven groups of the classification system should be taken, based on the domain of carbonaceous material around the crosswire which has the same optical texture as that of the marked point, **AND NOT ON THE WHOLE PARTICLE**.

It has to be stressed that these classes take into account all the possible occurrences of char textures but not all of them will necessarily be present in any particular char.

Results of the manual analysis:

The results of the BB2 (N) char microscopy analysis are shown in Figure 1. In the Figure are compared the results of the 1998 (red triangles) and the 1999 exercise (blue rhombi). The various participants acceptably agreed in the amounts of unfused material reported, and also in the amounts of fused-porous and fused-dense materials, the major problems being found in the amounts of isotropic and anisotropic material reported. In the slide session following the presentation of the results, some of the problems with the assignments were addressed, especially those dealing with how to classify fusinites exhibiting wavy-like anisotropy, which is likely to be original and not generated during the pyrolysis process. It was agreed that this material should be included in the group 7 corresponding to unfused fusinites. Some other topics discussed dealt with the criteria to distinguish between fused-dense and fused-porous inertinites. As experience shows that, after discussion of the micrographs, all participants tend to approach each other´s criterion, and as char preparation and distribution is rather expensive, it was decided to analyse for the 1999 Round Robin exercise the same sample, plus the one used by the combustion working group in their 1998 round robin exercise.

As in previous exercises, the spread of results was very large. The results only refer to 7 sets of data and therefore the statistic treatment is rather limited.

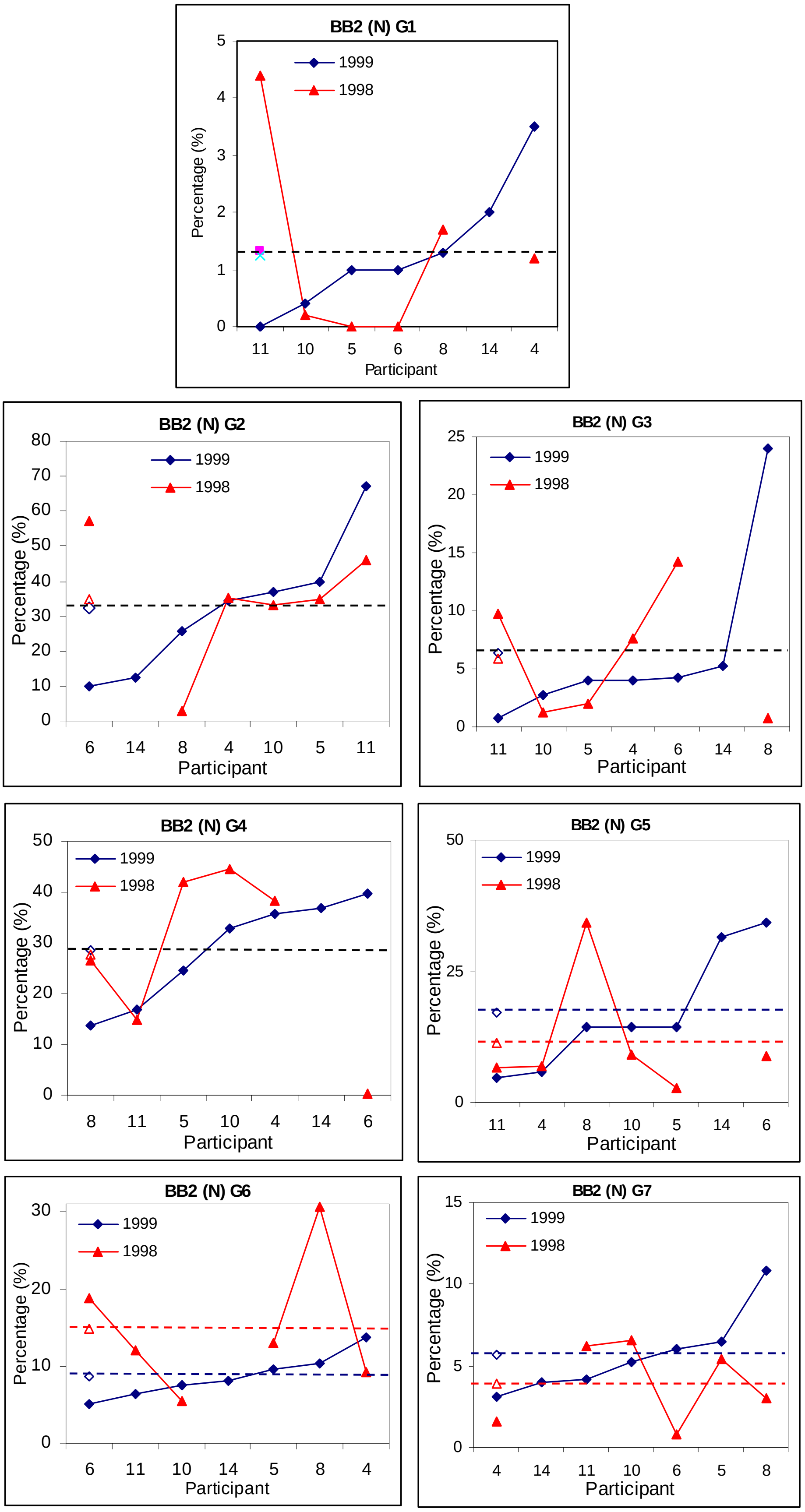


Fig.1. Results of the 1998 (triangle) and 1999 (rhomb) exercises on coal char BB2 (N). The dotted lines are the mean values.

Figures 1 and 2 show the results of the manual analysis for the two exercises in BB2 chars. Figure 2 indicates that the level of agreement among participants was good for G1 (vitrinite-derived material) and unfused material (G6+G7), but both the amount of porous *vs*. dense and isotropic *vs*. anisotropic concentrated the largest discrepancies.

The consistency of participants can be compared in Fig.2 in which results from 1998 exercise and 1999 exercise on BB2 char are shown. The comparison is necessarily limited since only participants 4, 5, 6, 8, 10 and 11 carried out both round robin exercises. Nevertheless these results indicate that participants 4, 5 and 10 reported similar results in both exercises, participant 8 reported similar amounts of porous and dense materials, but used a modified criterion for distinguishing isotropic and anisotropic material, and participants number 6 and 11 modified the criteria for distinguishing both the porosity and the anisotropy.

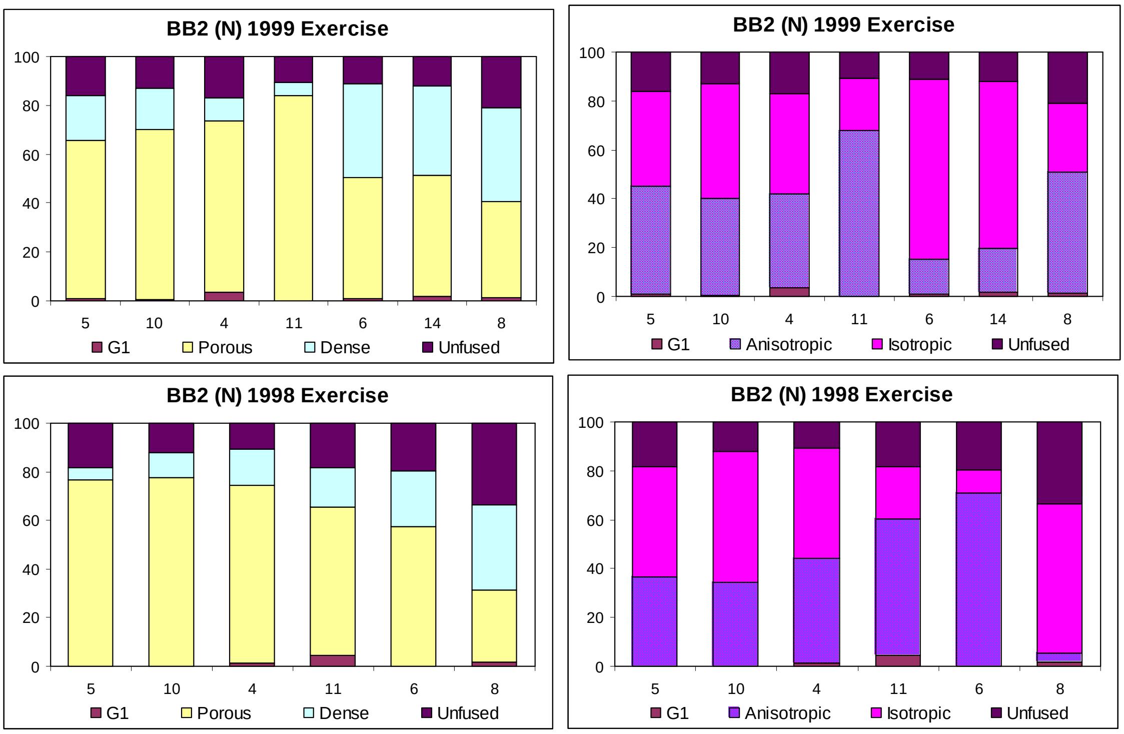


Figure 2. Plot of the results of the manual char analyses BB2 (N) Char. Anisotropic=G2+G3; Isotropic= G4+G5; Unfused=G6+G7; Porous=G2+G4; Dense=G3+G5.

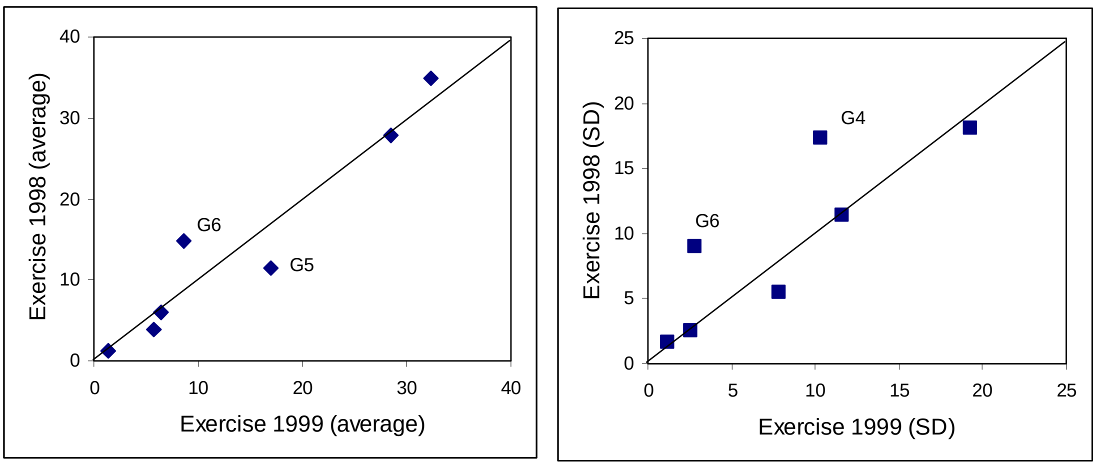


Figure 3. Plot of the average and standard deviation values for the different groups in the 1998 and 1999 exercises.

The plot in Figure 3 indicates that similar mean values were achieved for each char class in 1998 and 1999 exercises, the major differences being observed for G6 and G5 classes. Overall the standard deviation were similar or lower in the 1999 exercise indicating the favourable effect on the agreement of the discussion held at the meeting in Porto. Whereas in the amount of porous and dense material the participants were very consistent in both exercises in the amount of isotropic and anisotropic materials the scatter was very large. After the discussion held at the meeting in Porto the participants tended to report higher amount of anisotropic material than in the sample analysed the previous year.

As regards the behaviour of inertinite in a coal with Rr=066%, most of the inertinite developed porosity upon devolatilization (40-80%) and a significant part of it developed anisotropy (Figure 4).

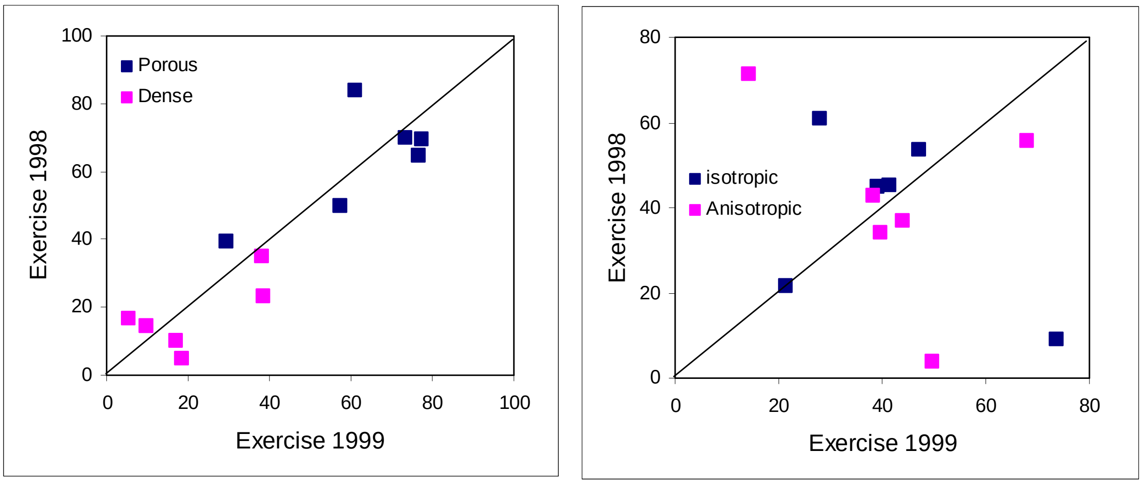


Figure 4. Amount of Porous and Dense and Isotropic and Anisotropic material reported by participants in the 1998 and 1999 exercises on coal char BB2 (N).

The results of Collinsville char (L) are shown in Figure 5 and the scatter of results is maintained within reasonable limits only for unfused material (G6 and G7). Despite the scatter, for classes G2 and G3 still are modal values observed and therefore the largest discrepancies are those affecting vitrinite-derived material (G1) and isotropic fused inertinite (G4 and G5).

If the results of porous/dense and isotropic/anisotropic are compared as in the case of BB2 (N), it is observed that a significant part of the inertinite developed porosity and also showed anisotropic optical texture. Vitrinite of Collinsville (L) coal generated anisotropic porous particles and therefore difficulties to discriminate between G1 and G2 could be expected. Indeed participants reporting high amount of G1 group reported low amounts of G2 group and vice versa (Figure 5). Despite the relatively high rank of the coal (Rr=1.28%) only a minor part of inertinite has been reported as unfused and this is a consistent result for all the participants.

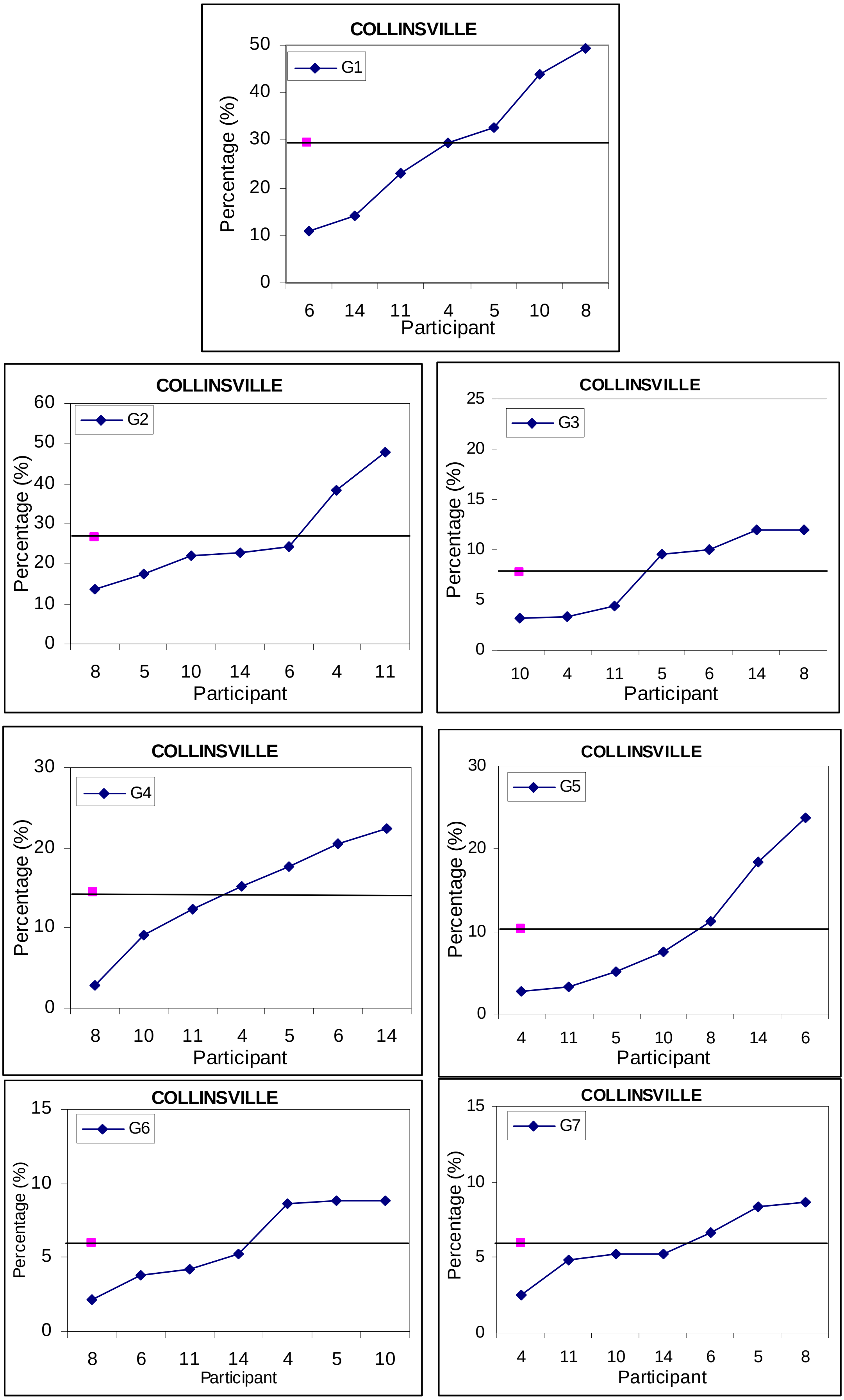
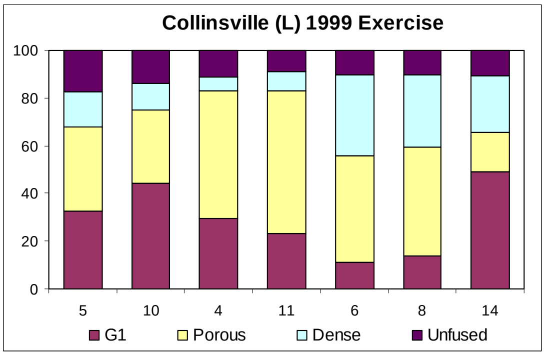


Figure 5. Results of the manual analysis of char Collinsville (L). Exercise 1999



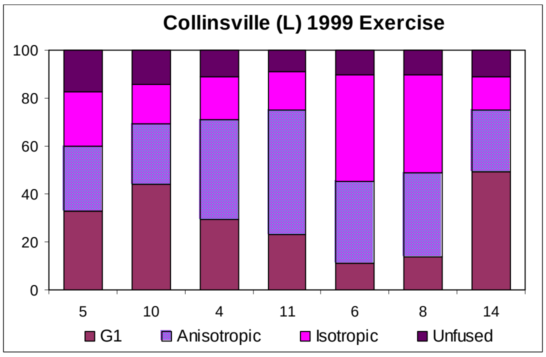


Figure 6. Plot of the results of the manual char analyses. Anisotropic=G2+G3; Isotropic= G4+G5; Unfused=G6+G7; Porous=G2+G4; Dense=G3+G5.

Considering the good results obtained on CD images (Borrego et al., INCWG 1999 CD) and the large scatter in manual analysis results (Borrego et al., INCWG 1997 and this report), most of the reasons for the discrepancies must be attributed to the variable magnifications used and to the observation conditions (i.e. use or not of retarder plate).

The results of the CD analysis also showed that both BB2 (N) and Collinsville (L) chars concentrated many dubious identifications and this is no doubt an additional reason for the scatter in the manual analysis results.

Suggestions for 2000 Round Robin exercise:

Considering the results summarised in this report, the suggestions for this year exercise are:

- Improve the definitions of the material to be classified within each group
- Focus on CD round robin exercises. Prepare an additional CD containing images taken with and without retarder plate in order to find out about the influence of observation conditions in the identifications.