Computer Assisted Rotated Reflectance Analysis of Vitrinite Macerals in Coal

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ABSTRACT

Rotated reflectance analysis of vitrinite macerals under the microscope is a valuable tool used by coal petrographers to determine the rank of coals. The analysis is subject to several sources of errors that the petrographer must be careful to avoid. This paper describes a computer aided analysis procedure that detects many errors that affect reflectance results during rotation of the specimen under the microscope.

INTRODUCTION

The maximum reflectance (Rø max) of vitrinite macerals obtained while rotating the microscope stage is used to rank coals and to predict the strength of cokes that can be made from coals (1). In recent years the bireflectance of vitrinite macerals has also gained importance to distinguish the difference between uniaxial and biaxial vitrinite reflectance (2).

The maximum reflectance value obtained from a vitrinite maceral while rotating the sample 360 ° can be affected by many sources of errors that include:

- air bubbles in the oil;
- contaminants in the oil such as lint;
- rotating too close to the edge of the maceral;
- rotating over a fissure or pit mark;
- rotating on non vitrinite macerals;
- fluctuations in the electronic signal from the photometer.

To avoid these errors,the petrographer must constantly observe the maceral during the rotated reflectance analysis which causes fatique and eye strain. CANMET's computer-aided rotated reflectance program simplifies and shortens the time required to collect the data for minimum and maximum vitrinite reflectance determinations. Operator eyestrain is reduced and accuracy of results are ensured while the ASTM standard is maintained (3). The software also calculates bireflectance; Rø max; draws histograms and bireflectance graphs; and provides data files for coke stability predictions.

EQUIPMENT

CANMET's microscopic system consists of a Zeiss Universal research microscope, a Zeiss photometer, a 12 volt 100 watt halogen light source, a Data Tech peak recorder, a 40 power oil immersion objective, a variable tube factor of 1.25, 1.65 and 2.0, 12.5 power eye pieces, a polariser set at 45°, a 546 nm filter and a 5 um limiting aperture. CANMET uses a MacIntosh Mac II computer with a MacADIOS II data acquisition board and an I/O daughter board installed for data acquistion from the microscope. A cable connects the signal from the microscope photometer to the I/O daughter board. A foot pedal connected to the data board allows the petrographer to signal the computer when to record readings. Software packages required to access the data acquisition boards are MacADIOS Turbodrivers and MacADIOS Data Manipulation Library.

PROGRAM

The program displays readings on the computer screen during standardization of the photometer. To increase speed and accuracy the electronic signal from the photometer is sent to the MacADIOS' analog to digital (AD) converter instead of to the AD converter of the microscope. The computer takes 6000 readings during one 360° rotation of the stage for each vitrinite maceral. From these readings the following information is calculated, recorded to file and displayed on screen:

- minimum and maximum readings;
- bireflectance;
- average of the minimum and maximum readings;
- average of the 6000 readings;
- difference between the two averages.

At the completion of the analysis the computer displays the mean Rø max for every 100 vitrinite maceral determinations and for the entire sample. The percentages of vitrinite found in each half V-type are stored in data files that can be used directly by commercial software programs to produce histograms, graphs and statistical analysis. The files are also used by another software program to predict ASTM coke stabilities. Figure 1 shows values for minimum and maximum reflectance for a coal plotted against bireflectance. Figure 2 shows an example of the computer output for vitrinite distribution in a coal and a histogram of the vitrinite reflectance data based on half V-type.

An alarm warns the operator if the difference between the average of the 6000 readings and the average of the minimum and maximum readings for one rotation, is greater than 0.04. The operator is also warned if the bireflectance is greater than a preset level. A bireflectance limit is preset for every coal which can be as low as 0.2 for low rank coals and as high as 0.4 for high rank coals. This limit must be set (and sometimes reset during analysis) for each coal so that it is always slightly higher than the highest valid bireflectance reading obtained on vitrinite. If no source of error can be detected after a warning alarm is sounded and repeat rotations give the same results, then the bireflectance limit is set too low.

RESULTS

Table 1 is an example of valid readings for two different vitrinite macerals in the same coal. The difference between the average of the 6000 rotated readings and the average of the minimum and maximum readings is very consistent even though the rotation was taken at different locations on the macerals. The bireflectance also remains consistent for each of these vitrinite macerals. Consequently, errors can be detected readily by computer analysis because of the consistencies of these data and because valid bireflectance readings will always fall below correctly set limits.

Table 2 shows reflectance data obtained when the following sources of errors occurred during the analysis: bubbles, edge effect, fissures and pit marks, boundaries, and contaminants in the oil respectively. The bireflectance limit for this coal was set at 0.25. The bireflectance for all of these conditions is consistently (and considerably) higher than the set limit. The difference between averages is not as reliable in detecting these sources of errors and is used mainly to detect electronic fluctuations which, although rare, do occur..

Table 3 shows that reflectance data collected by rotating the stage on different semifusinite macerals also can set off a warning to the operator. In general the bireflectance value for a semifusinite maceral is higher than for a vitrinite maceral and would exceed the preset set limit. Although the computer alarm would indicate one of the previously mentioned errors, the high bireflectance in this case is caused by the semifusinite maceral itself.

Table 1 Valid rotated reflectance readings of two vitrinite macerals

	Rø max	Rø min	Avg Rø	Biref*	Diff**
Maceral 1	1.42	1.24	1.33	0.18	0.00
	1.42	1.27	1.34	0.15	0.00
	1.43	1.26	1.35	0.17	0.01
	1.43	1.26	1.34	0.17	0.00
	1.43	1.27	1.35	0.16	0.00
	1.43	1.25	1.34	0.18	0.00
	1.41	1.27	1.33	0.14	0.01
Maceral 2	1.42	1.32	1.37	0.10	0.00
	1.41	1.31	1.36	0.10	0.00
	1.42	1.30	1.36	0.12	0.00
	1.43	1.30	1.36	0.13	0.00
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.37	0.11	0.01
	1.42	1.31	1.37	0.11	0.01
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.36	0.11	0.00
	1.42	1.31	1.37	0.11	0.01
	1.42	1.30	1.37	0.12	0.01
	1.42	1.31	1.36	0.11	0.00

* Bireflectance (the difference between the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral)

** Difference between the average of the 6000 readings and the average of the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral.

Table 2 - Sources of errors for non-valid vitrinite reflectance readings

	Rø max	Rø min	Avg Rø	Biref*	Diff**
Air bubbles	1.40	0.01	0.82	1.39	0.12
	1.38	0.01	1.07	1.37	0.38
	1.39	0.01	0.71	1.38	0.01
	1.38	0.01	0.76	1.37	0.07
	1.36	0.01	0.55	1.35	0.13
	1.36	0.01	0.47	1.35	0.21
	1.40	0.23	1.02	1.17	0.21
	1.40	0.34	0.98	1.06	0.11
	1.41	0.39	0.91	1.02	0.01
	1.42	0.34	0.97	1.08	0.09
	1.34	0.12	0.83	1.22	0.10
Edge effect	1.57	1.10	1.44	0.47	0.11
	1.58	1.02	1.44	0.56	0.14
	1.57	1.18	1.44	0.39	0.07
	1.58	1.05	1.47	0.53	0.16
Fissure/pitting	1.55	1.12	1.37	0.43	0.04
	1.56	1.16	1.39	0.40	0.03
	1.56	1.12	1.41	0.44	0.07
	1.56	1.15	1.43	0.41	0.08
	1.57	1.14	1.44	0.43	0.09
	1.56	1.24	1.42	0.32	0.02
Maceral/resin boundary	1.53	0.26	1.07	1.27	0.18
	1.52	0.26	1.01	1.26	0.12
	1.53	0.26	1.01	1.27	0.12
	1.53	0.26	1.01	1.27	0.12
Contaminants in immersion oil	1.73	1.26	1.35	0.47	0.14

* Bireflectance (the difference between the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral)

** Difference between the average of the 6000 readings and the average of the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral.

Table 3- Reflectance data obtained for semifusinite macerals

Rø max	Rø min	Avg Rø	Biref*	Diff**
1.92	1.37	1.70	0.55	0.06
1.75	1.28	1.55	0.47	0.04
1.82	1.31	1.54	0.51	0.02
2.19	1.77	1.97	0.42	0.01
1.80	1.32	1.67	0.48	0.11
1.98	1.41	1.68	0.57	0.01

* Bireflectance (the difference between the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral)

** Difference between the average of the 6000 readings and the average of the minimum and maximum readings obtained for one 360° rotation on a vitrinite maceral.

CONCLUSIONS

By comparing bireflectance values against preset limits a computer program has been developed to detect errors during rotated reflectance analysis of vitrinite in coal. The sources of errors can be caused by air bubbles, rotating too close to the edge of a maceral, fissures, pit marks, boundaries and oil contaminants. The program increases the accuracy and the speed of analysis and minimizes the number of repeat analyses. Because the program relieves the operator of the responsibility of detecting most errors, eye strain is reduced and productivity improved. Petrographers using this program have greater confidence in the validity of their reflectance data because errors that are normally hard to identify are easily detected.

ACKNOWLEDGEMENTS

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(3) Standard Test Method for Microscopic Determination of the Reflectance of the Organic Components in a Polished Specimen of Coal, D 2798-88, 1991, Annual Book of ASTM Standards, section 5, vol. 05.05, pp. 312-314.

Lab number	: 0123-62
Project number	: 03-3-1/6-33
Company name	:
Pellet number	: 2943
Based on	: 100 Reflectance Readings

V_types	%	V_<0.49	%	Avg.	V_>0.50	%	Avg.
V_ 10 :	2.0%	V_10.00:	0.0%	0.000	V_10.50:	2.0%	1.075
V_ 11 :	13.0%	V_11.00:	4.0%	1.125	V_11.50:	9.0%	1.172
V_ 12 :	26.0%	V_12.00:	11.0%	1.218	V_12.50:	15.0%	1.277
V_ 13 :	36.0%	V_13.00:	23.0%	1.314	V_13.50:	13.0%	1.367
V_ 14 :	17.0%	V_14.00:	11.0%	1.423	V_14.50:	6.0%	1.472
V_ 15 :	6.0%	V_15.00:	6.0%	1.507	V_15.50:	0.0%	0.000

Average Rø max per 100 readings and Mean Rø max

1st Set	2nd Set	3rd Set	4th Set	Mean Rø
1.32	1.31	1.30	1.32	1.31

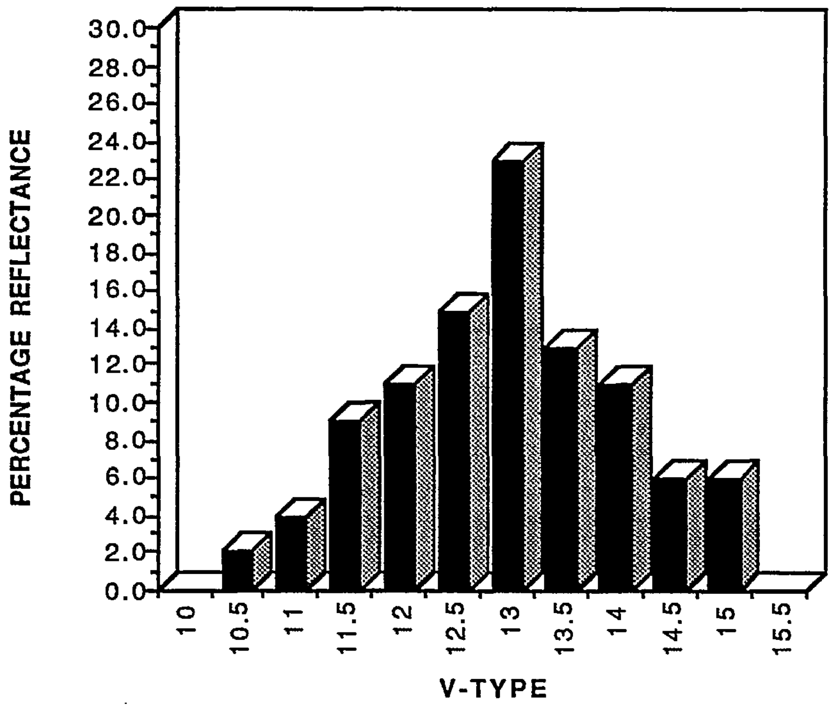

Fig. 2 Vitrinite reflectance distribution and histogram of a coal sample

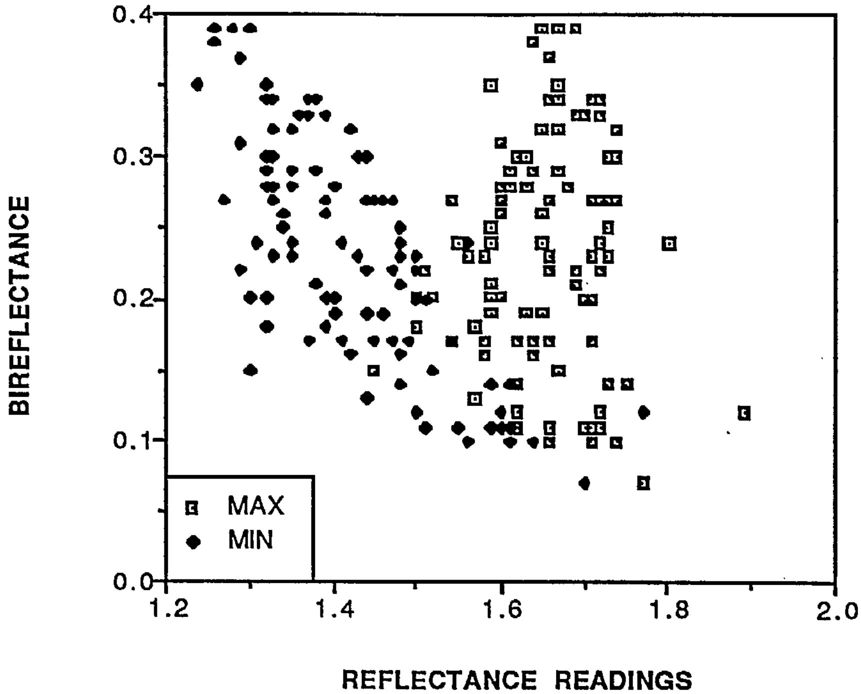

Fig. 1 Plot of vitrinite bireflectance versus minimum and maximum reflectance.