Characterizing Galvanizing Bath with Fluorescence

Introduction

Automobile-parts production can be paralyzed by a defective electron-galvanizing (EV) bath line. Because a sensitive and accurate analysis of EV solutions is fundamental to productivity, quality, and identification of errors, a detailed analytical profile can help trace the source of error and contribute to selecting the best solution. Both down-time and material cost therefore can be minimized.

Fluorescence spectroscopy is an analytical technique with high sensitivity and selectivity. By characterizing the components in the EV-coat solution with fluorescence, a detailed analytical profile can be formed for each component. This can help trace the source of errors in the EV-coat solution, and contribute to selection of the best formulation. Quality-control tests can be performed periodically to check for consistent bath composition.

Instrumentation

The FluoroMax® bench-top spectrofluorometer was used in this investigation. This system is compact, economical, and offers many automated accessories. The FluoroMax® is noted for its outstanding sensitivity, speed, and easy-to-use software.

- High sensitivity via all-reflective optics and photon-counting detection.
- Data-acquisition is fast because the system can slew at 80 nm s^{-1}. Coupling sensitivity and speed means efficiency and productivity in the laboratory.
- Complete experiments can be stored and recalled for reproducibilty.

Therefore, spectra can be acquired in seconds, and a time-based scan can be collected at 1 ms/data point. Simplicity is apparent in the automation, computer-controlled variable slits, software, and calibration.

Experimental method

Samples obtained for analysis were divided into six segments. EV bath was a sample of the current bath causing problems for the operator. Sample A is the plating bath that would compose the ideal EV bath. Sample B is a bath that contains 0.6 mL of starter and 1.5 mL of brightener. Sample C is a bath that contains 2 mL of starter and 0.5 mL of brightener. Starter and brightener stock samples were obtained for comparison.

Results and discussion

The six samples were characterized by their excitation and emission spectra (Figs. 1–4). The fluorescence spectra may offer a method for correctly mixing the appropriate concentrations or proportions of starter and brightener solutions in the EV bath. For the coating process to work, a constant level of starter and brightener is necessary.

Fig. 1 illustrates that sample A (the ideal bath) was 5×10^5 counts lower in fluorescence intensity than the EV bath. Sample C was 10^6 counts higher in intensity than the EV bath. Therefore, a component in sample C must have been the cause of the greater intensity. Sample C contained the largest amount of starter solution, and the smallest volume of brightener solution. This indicates that the actual EV bath

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contained too much starter solution, based on the difference in fluorescence intensities.

The data were fitted to the following equation:

$$I_f = A[\text{Brightener}] + B[\text{Starter}] + C$$

where A, B, and C are constants, [Brightener] is the concentration of brightener, [Starter] is the concentration of starter, and I_f is the observed fluorescence intensity. An empirical linear relationship thus was established between the fluorescence intensity and ratio of brightener to starter:

$$[\text{Brightener}] = \frac{[\text{Starter}] + 2.7 - I_f}{1.4}$$

This equation is merely a simple form of chemometrics. Larger, more complex experimental analyses can be handled with the use of a more elaborate form of chemometrics.

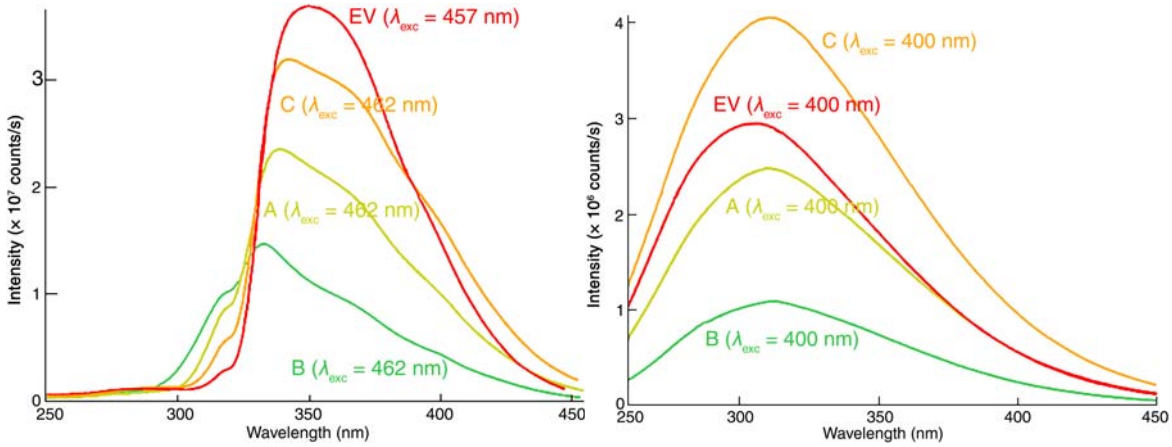

Fig. 1. *Excitation (left) and emission (right) spectra of Samples A, B, and C, and EV bath. Excitation or emission wavelength is given next to each trace.*

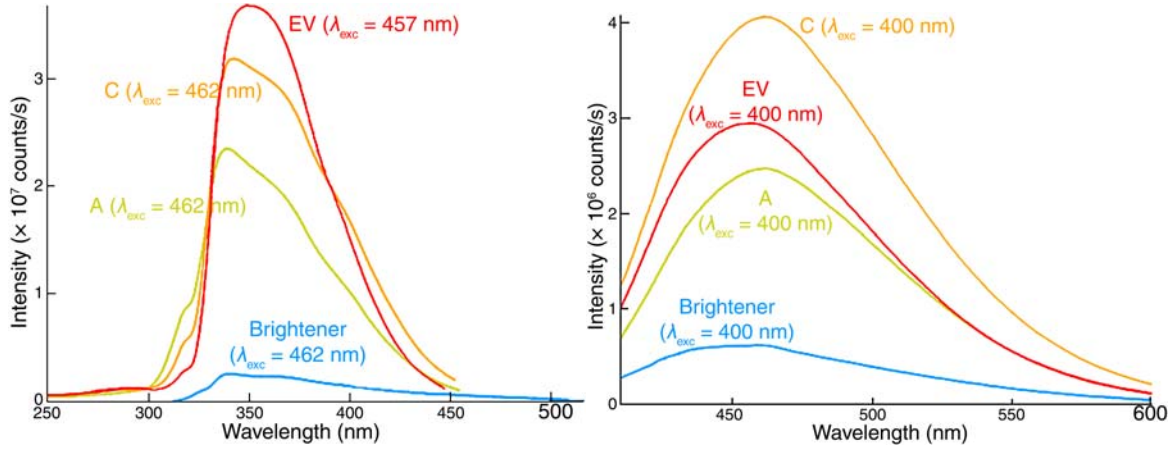

Fig. 2. *Excitation (left) and emission (right) spectra of Samples A, C, EV bath, and brightener. Excitation or emission wavelength is given next to each trace.*

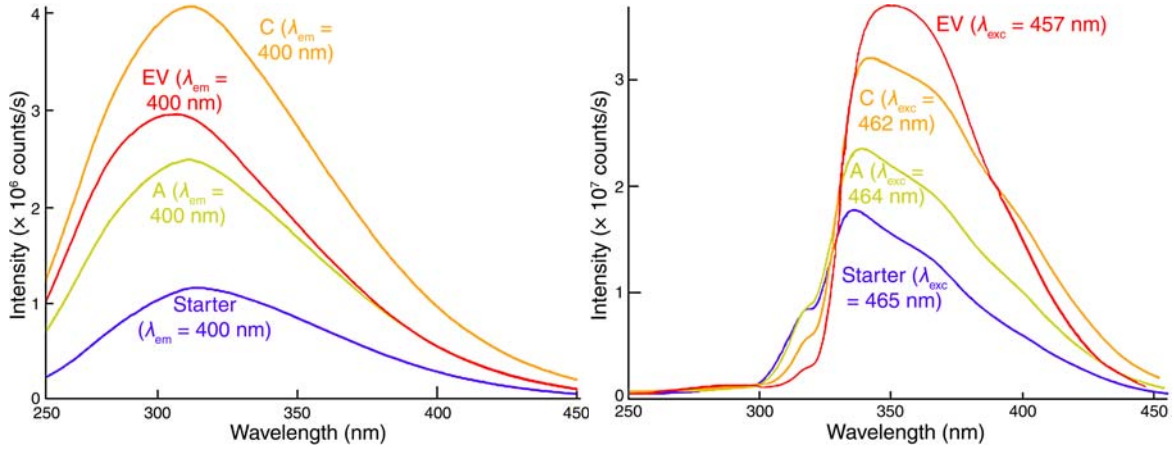

Fig. 3. *Excitation (left) and emission (right) of Samples A, C, and a 100-fold dilution of starter solution. Excitation or emission wavelength is given next to each trace.*

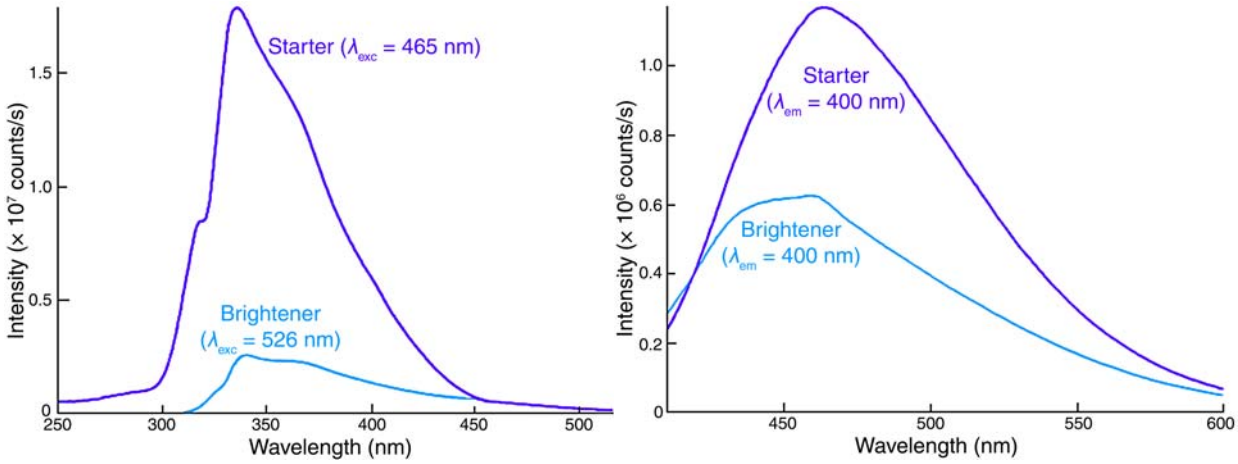

Fig. 4. *Excitation (left) and emission (right) spectra of of stock solution of brightener and 100-fold dilution of starter. Excitation or emission wavelength is given next to each trace.*

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