This manuscript describes an interesting study combining EEMS and FT-ICR-MS analysis of SPE-extracted DOM samples from a black-water, a white-water, and a clear-water river within the Amazon basin. As such it provides one of the most detailed compositional studies of the DOM within a water system to date and provides key information to add to the large body of work on bulk organic matter characteristics within the Amazon Basin. The topic and general scientific approach make the study a good one for publication in this journal. The problem with the manuscript right now is that it appears to have been written assuming that the reader will know all the details of the analytical techniques and multivariate analyses. Key information is either missing or presented but not explained.

General comments: In Section 2.2., the limits of detection, and a measure of precision and accuracy need to be given for each technique. In section 2.3, the authors need to tell the reader whether positive or negative ionization is used and what solvent the samples were brought up in and what the mobile phase was, if there wasn’t direct injection of the sample. The details promised in the supplementary file are not present. In Figure 1 (representative FT-ICR-MS spectra) and in Figure S2, the reader needs to know which samples these were, mainstem or some other location. Perhaps the stations in Figure S1 could be given individual names or numbers and these could be used in the captions to Figure 1 and S1? Three panels in Figure 3 have $-\text{KMD}/z^*$ as the $y$ axis but the reader is never told what this means (I assume a Kendrick mass defect factor) nor are these panels discussed anywhere in the text or supplemental section. Finally, the description of the heat maps needs to be revised for clarity. I tried very hard through multiple readings of the text (with the figures printed out for quick reference) to understand exactly what was being correlated in each figure but was unable to do so. Could there be some guidance along the $x$ and $y$ axes and more description in the text of the technique itself as well as the results?

Specific comments:

1. The superscripts throughout this manuscript appear to have been lost in a formatting step.
2. Page 2, line 12, “has” should be “have”
3. P.3, line 11, “exists” should be “exist”
4. P. 4, line 7, should read “Small (40-mL) aliquots”
5. P. 4, line 18. Which convention should be used in this journal for five hundred thousand, the decimal or comma form?
6. P. 4, line 30. “One hundred microliters”?
7. Figure 1 is mislabeled in its caption as Figure 2.
8. P. 6, lines 3-6. This sentence is trying to say too many things and I cannot follow the points. Please reword. Perhaps start by pointing out the formulae for your hydrogen deficient vs more saturated compounds
9. P. 6, line 13, “where” should be “were”
10. P. 7, line 3, what does “disk-shaped” mean? You cannot know higher order folding of the molecules from MS data, can you? Or are you referring to a disk-shaped distribution of formulae in the van Krevelen?
11. P. 7, line 29, “close” should be “closely”
12. P. 7, line 31, “high” should be “highly”
13. P. 8, line 10, should read “long-wavelength absorbance”
14. P. 8, lines 8-13. I am not sure I am following this reasoning. Please clarify.
15. P. 8, lines 14-16. Please reword this sentence for better clarity.
16. P. 8, line 27, add “of” before “which”
17. Please add information on the color scale and axis or variable labels for the heat maps.