CAN BE USED DSC TO STUDY PET BOTTLES?

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Introduction

In this study, we focused on verifying that the use of differential scanning calorimetry (DSC) can lead to the identification of polyethylene terephthalate (PET) bottles with a specific composition.

As some studies suggest, PET properties depend on its microstructure and are determined by crystallization rate, the degree, and quality of crystallinity. DSC parameter (glass transition, crystallization, and melting behavior of PET) depend on crystallinity, which varies depending on the use of different feedstock types. Hence, the parameters from DSC should correspond to the PET composition.

In general, the types of bottles investigated in this work can be divided into three groups according to their history. The largest group are bottles made entirely of virgin PET. The second group contains bottles containing additives, which improve barrier properties. However, these additives act as contaminants during recycling. Therefore, it is necessary to find a way to identify them and find a way to exclude them from the recycling stream. The third group represents bottles that contain a certain amount of recycled material – recycled PET (rPET) or PET made from natural sources (bioPET). PET in this group is currently of great interest due to European Union plans that will oblige producers to use these types in PET bottles. Besides, declaration of use rPET/bioPET in bottles is already a marketing advantage due to some consumers' preference. However, the proof of rPET/bioPET in a PET bottle is a great challenge.

Methods

Thermal properties of PET were analyzed using differential scanning calorimetry (DSC) TA Instrument DSC 2500 with a repeating thermal heating and cooling cycles of 5°C/min from 30°C to 320°C. The second heating and cooling were assessed to determine the parameters of glass transition, melting, crystallization temperature and enthalpy. These parameters were then statistically elaborated by two statistic approaches.

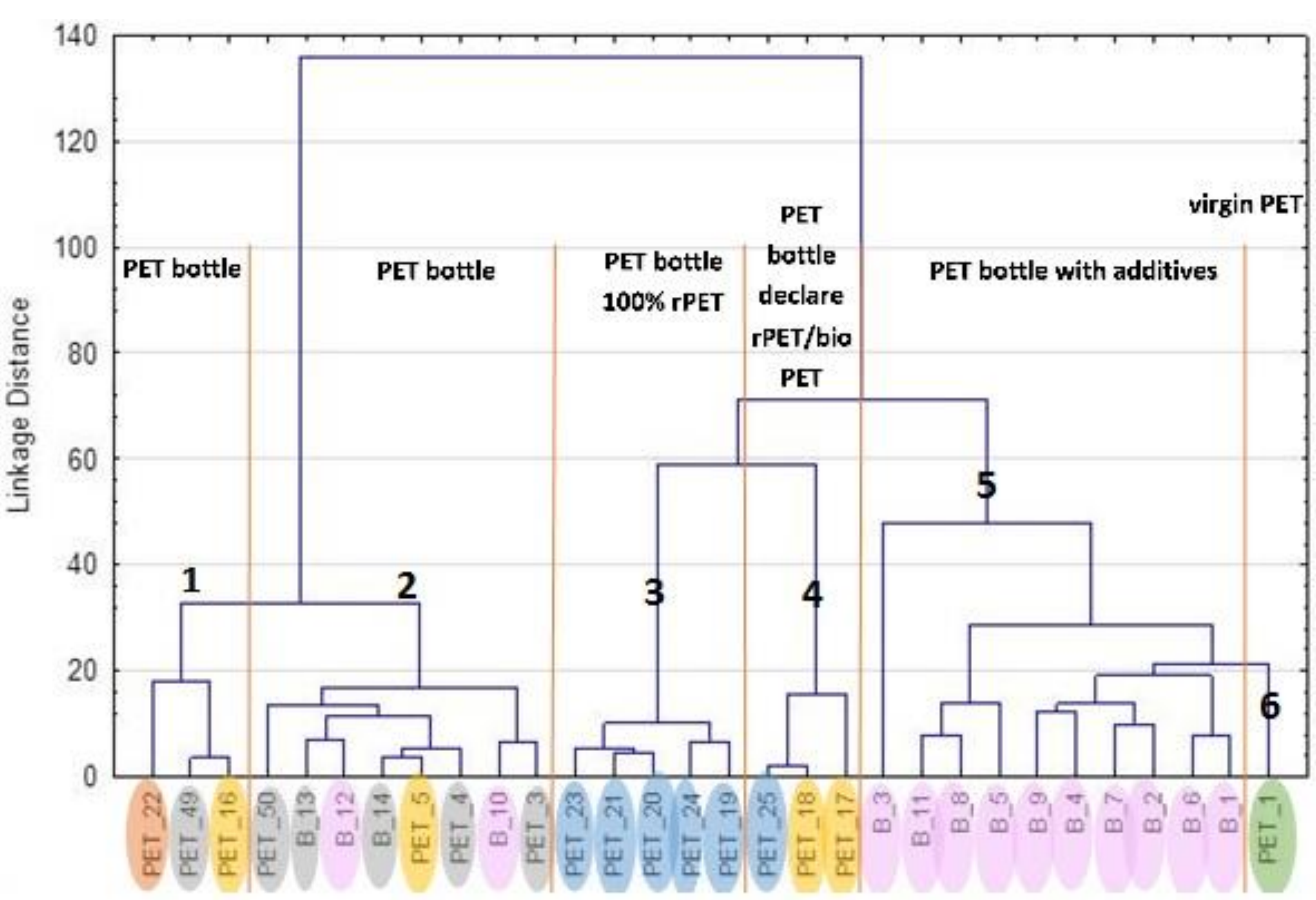
Samples

29 PET bottles and 1 control sample of PET virgin were analyzed. Bottles can be divided in to 3 groups of samples: PET bottles from virgin PET, PET with additives and PET bottles content rPET/bioPET

Group	Samples
PET virgin	PET_1
PET bottles from virgin	PET_3, 4, 49, 50; B_13, 14 (control to B_11, 12)
PET bottles with additives	B_1-B_12
PET bottles 100% rPET	PET_19-25, PET_22 - refill
PET bottles % rPET	PET_17, 18
PET bottles % bioPET	PET_5, 16,17

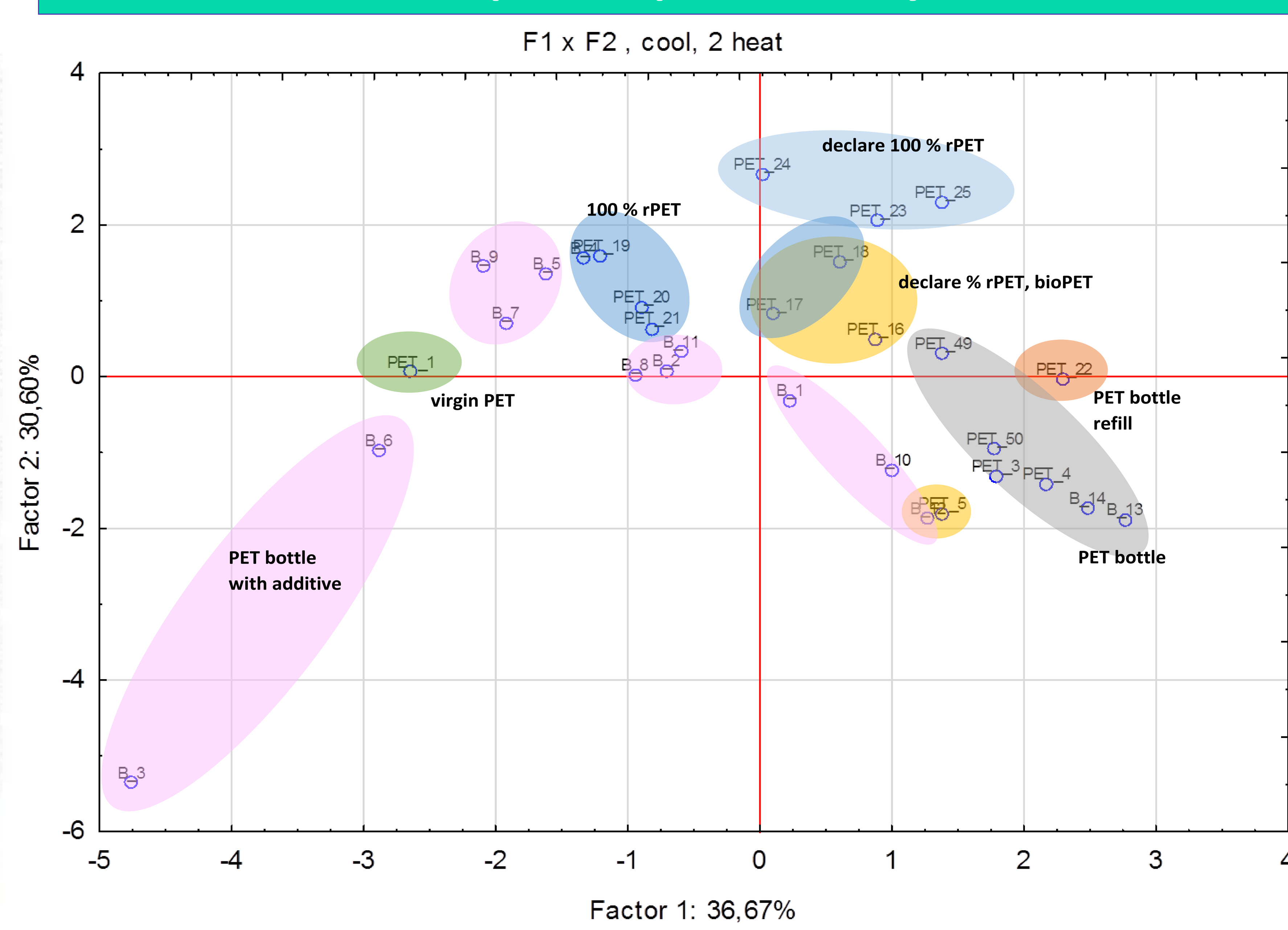
Results and discussion

Tree diagram "Ward's Method" (Euclidean distances)



- Data from the first and second heating and cooling were used for the Ward method. 6 clusters can be distinguished.
- The contents of clusters 1 and 2 mainly includes PET bottles and refill bottles and are thus reliably identifiable.
- All 100% rPET bottles belong to cluster 3 and are thus reliably identifiable. Bottles with a lower rPET / bioPET content belong to cluster 4. PET_25 is declared as 100% rPET, but probably contains less than 100% rPET. PET_5 and PET_16 (lower bioPET content) should be in cluster 4. But due to inclusion in clusters 1 and 2, the bioPET content is questioned.
- All PET bottles with additives belong to group 5 and are thus reliably identifiable. B_10 and B_12 are in cluster 2. They probably do not contain enough additives to prove.

Principal component analysis



- Data from the second heating and cooling were used for PCA analysis.
- PET bottles form a coherent group in the opposite quadrant than virgin PET. This group also includes a refill bottle (PET_22). This is in agreement with the first analysis. They are therefore reliably identifiable.
- PET_19-21 (100% rPET) forms a group in 1st quadrant. This division is confirmed by the first analysis. However, PET_23-25, declared as 100% rPET, do not belong to this group. Their location corresponds more to bottles with smaller content of rPET/bioPET (PET_17-18). Thus, content of 100% rPET is highly unlikely. This division is confirmed by the first analysis.
- PET_5 and PET_16 are declared as containing bioPET. But according to both analyzes belong more to PET bottles from virgin. Thus, content of bioPET is highly unlikely.
- Although in the first analysis, PET samples with additives belong to one cluster, in PCA they do not have a uniform position. This may be due to the use of different additives. Even so, it is at least partially possible to distinguish them.

Conclusions

The results suggest that the combination of DSC data and advanced statistical methods can divide analyzed bottles into these three categories.

- Bottles from virgin PET shown similar properties and can easily be identified.
- PET bottles with additives are a challenge due to different additives, but there is potential for their identification.
- Some bottles declared to contain rPET/bioPET may, in fact, not contain these PET types.

We conclude that DSC has a great potential to become a robust method for identifying PET bottles' different chemical compositions.

The financial support of project FCH-S-21-7398 of the Ministry of Education, Youth and Sports is acknowledged.