Microwave-assisted methods for greener and more practical (blueness) fatty acid profiling in food

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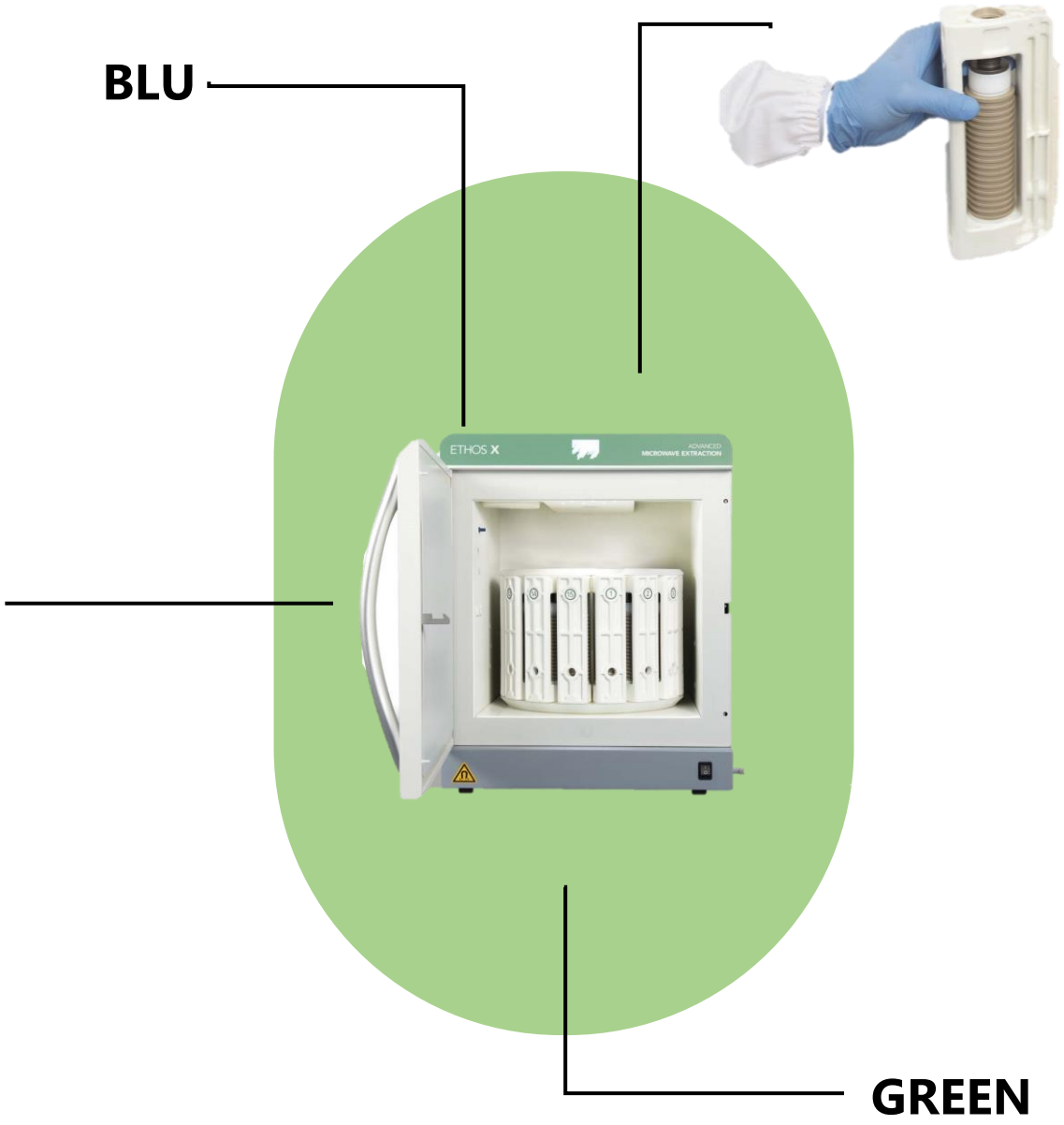
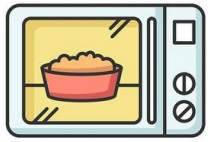


MICROWAVE TECHNOLOGY IN SAMPLE PREPARATION



1

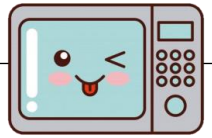
1945

DISCOVERY OF MICROWAVES

2

1947

FIRST MICROWAVE OVEN WAS SOLD



3

1975

FIRST DOMESTIC MICROWAVE OVEN USED FOR THE EXTRACTION OF ORGANIC COMPOUNDS

5

1993

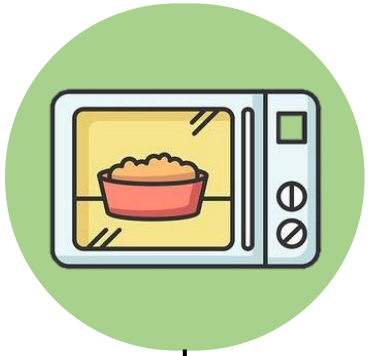
LABORATORY MW EXTRACTOR SYSTEM

6

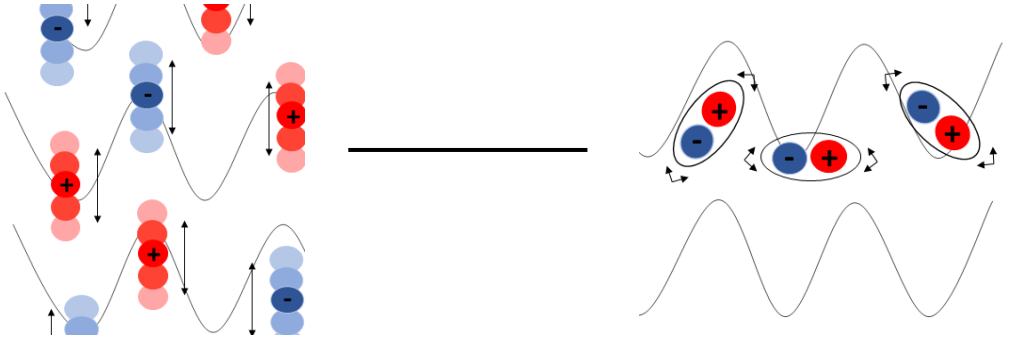
2024-

DEVELOPMENT OF NEW TECHNOLOGIES

HOW MICROWAVES HEATING WORK?



Microwave radiation refers to electromagnetic radiation spanning the 300 MH-300 GHz frequency range

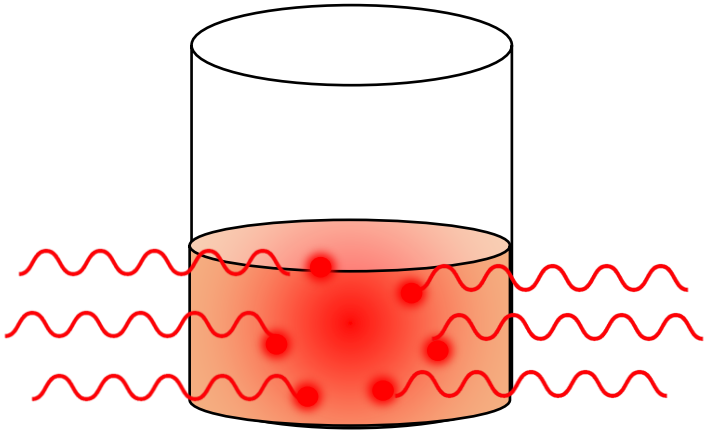


ION CONDUCTION

DIPOLE ROTATION

ADVANTAGES OF MICROWAVE HEATING

- Direct Heating
- High efficiency
- Short processing times
- Less Heating Exposure
- Green Process



WHY FATTY ACID ANALYSIS IS IMPORTANT?

FATTY ACIDS

Fatty acids (FAs) profiling relates to an analytical approach capable of providing detailed information about the quality-quantitative distribution of FAs in many food products.

CHARACTERIZATION

CHARACTERIZATION AND DETERMINATION OF TOTAL FAT CONTENT IN FOODS

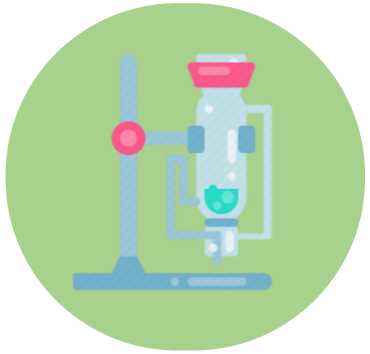
LABELING

FOR NUTRITIONAL LABELING AND TO UNDERSTAND THE AVAILABILITY OF FAT

HEALTH IMPACT

HEALTH IMPACT AND CORRELATION WITH SOME DISEASES

MICROWAVE TECHNOLOGY FOR FATTY ACIDS ANALYSIS



TRADITIONAL PROCEDURE

EXTRACTION OF LIPID FRACTION

DERIVATIZATION

GC-FID

ONE-STEP MICROWAVE-ASSISTED EXTRACTION AND DERIVATIZATION (MAED)

SIMULTANEOUS EXTRACTION AND DERIVATIZATION

GC×GC-FID

Fina, A.et al. **Advances in Sample Preparation 4 (2022) 100039**

AIM OF THE PROJECT

BEFORE

COMPARISON OF
MAED AND
OFFICIAL METHODS
AOCS Ce 2b-11

FIRST GOAL

AFTER

COMPARISON OF
MAED AND
OFFICIAL METHODS
AOCS Ce 2b-11
AOCS Ce 2c-11

AIM OF THE PROJECT

BEFORE

MANY LABORATORIES PERFORM THE MICROWAVES-ASSISTED EXTRACTION BUT THEN THEY **DERIVATIZE WITH BF_3**

Step 1
EXTRACTION

Step 2
DERIVATIZATION WITH BF_3

SECOND GOAL

AFTER

EXPLORE THE USE OF MICROWAVE TO PERFORM THE **DERIVATIZATION**

Step 1
EXTRACTION

Step 2
DERIVATIZATION with HCl/MeOH

EXPERIMENTAL PART

TWO STEP

Microwave-assisted Extraction

EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

HEXANE EXTRACTION

TOTAL FAT

Derivatization with **BF_3**

Derivatization with **HCl/MeOH**

ONE STEP

AOCS Ce2c-11

Direct Methylation by Acid- Alkaly hydrolysis

AcMeOH + NaOH + **BF_3** + Hexane

AOCS Ce2b-11

Direct Methylation by Alkaly hydrolysis

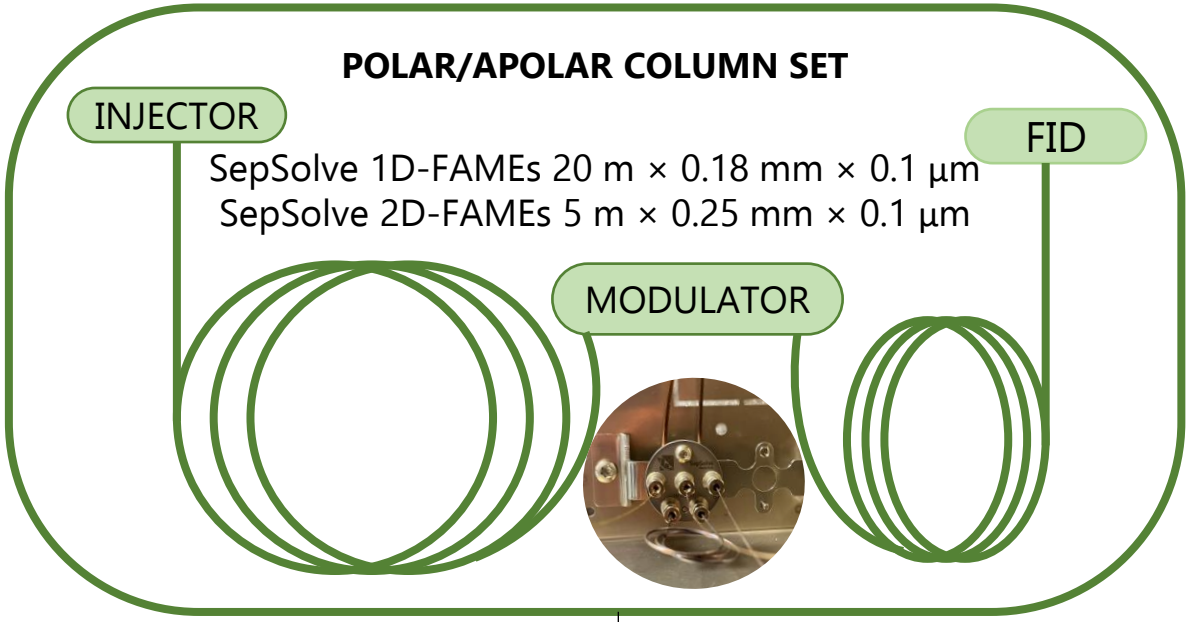
NaOH + **BF_3** + Hexane

MAED

One-Step Microwave-assisted extraction and derivatization
HCl/MeOH + Cyclohexane

EXPERIMENTAL PART- INSTRUMENTAL ANALYSIS

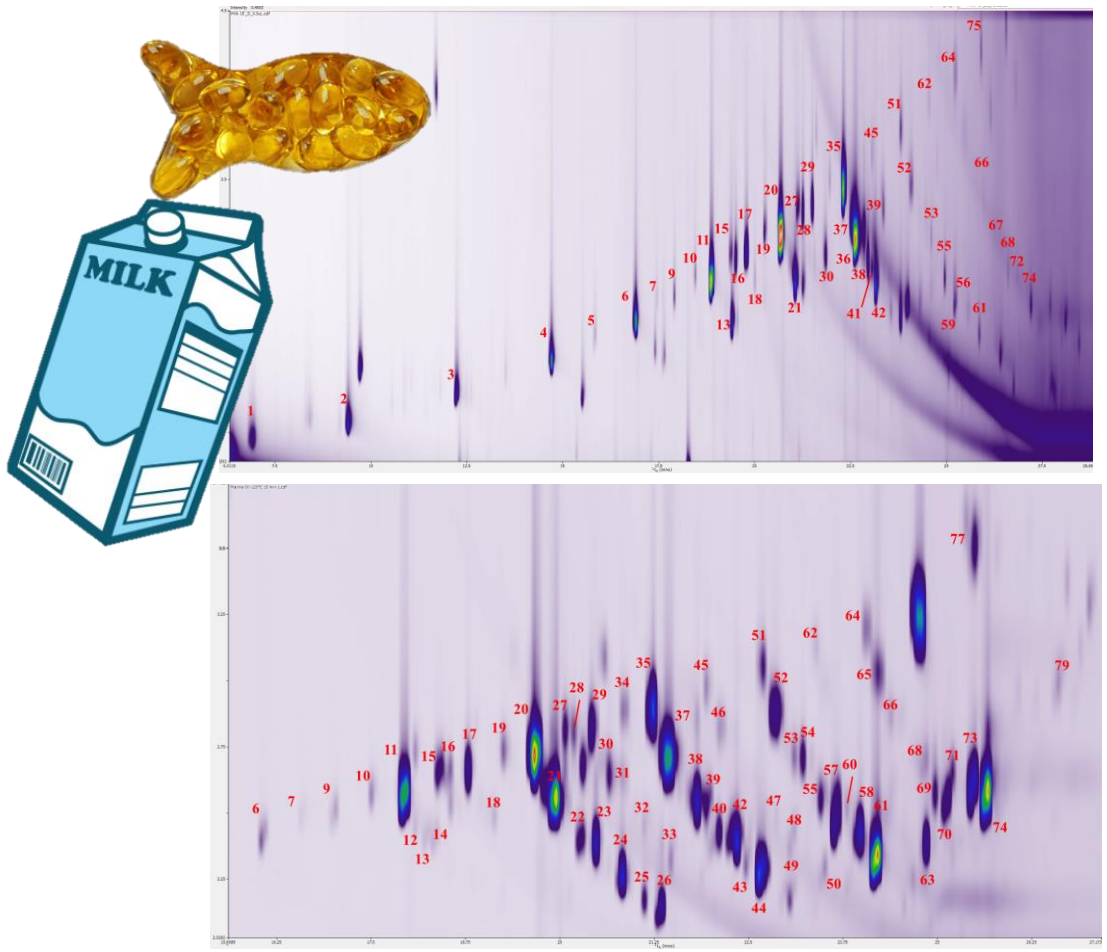
GC×GC-FID



ADVANTAGES

- ENHANCED SEPARATION
- REDUCED CO-ELUTION
- INCREASED SENSITIVITY
- STRUCTURED CHROMATOGRAMS

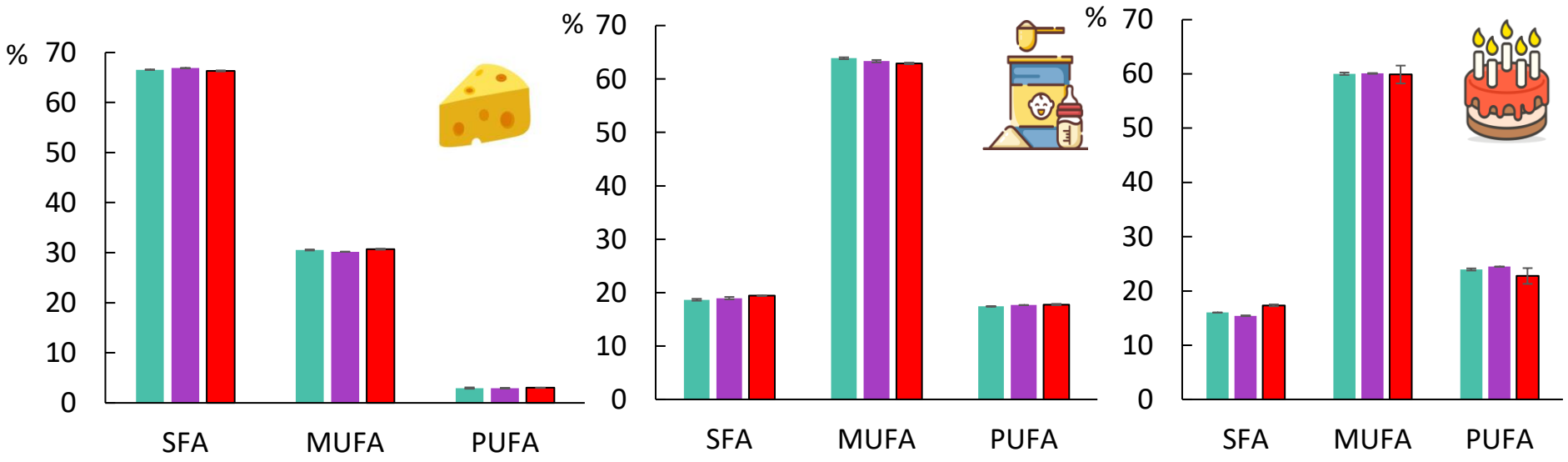
COMPLEX MATRIX



~ 30 min run

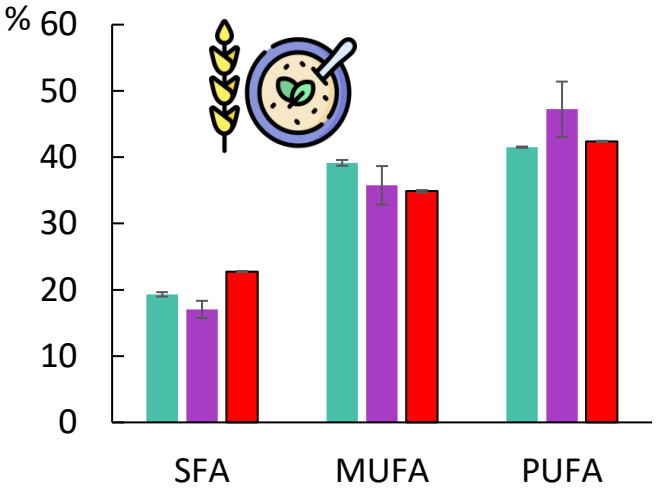
RESULTS

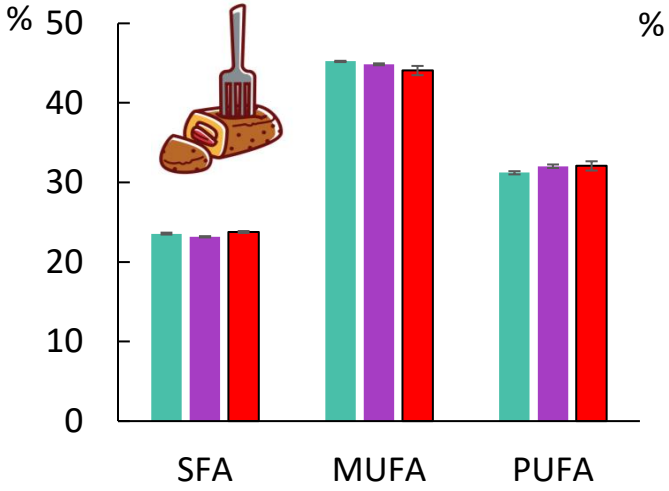
Official methods vs MAED

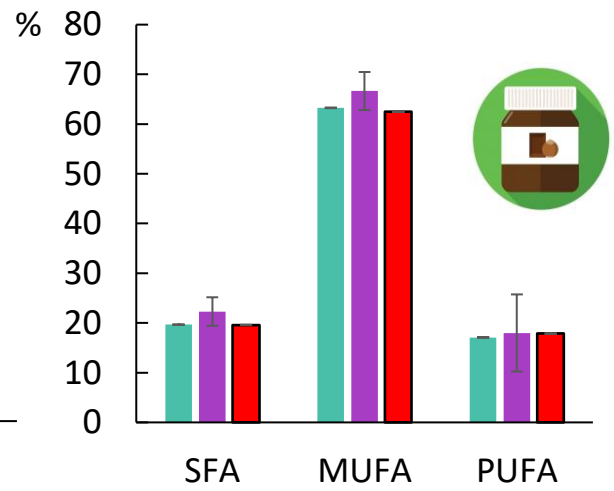


AOCS Ce2c-11

AOCS Ce2b-11





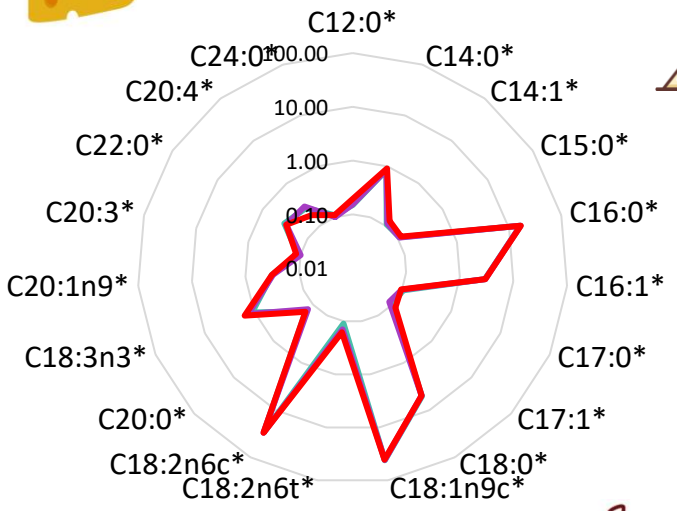


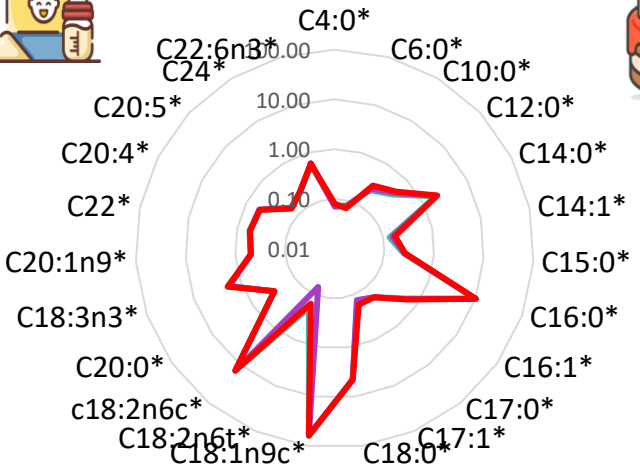
MAED

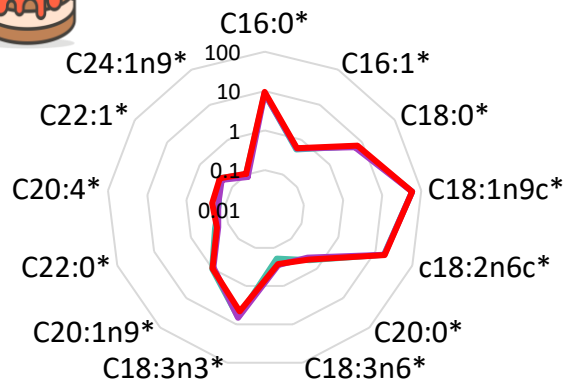
RESULTS

Official methods vs MAED

EXCEPT FOR THE OAT, WE CONFIRM THAT THE MAED IS A RELIABLE ALTERNATIVE TO THE OFFICIAL METHOD

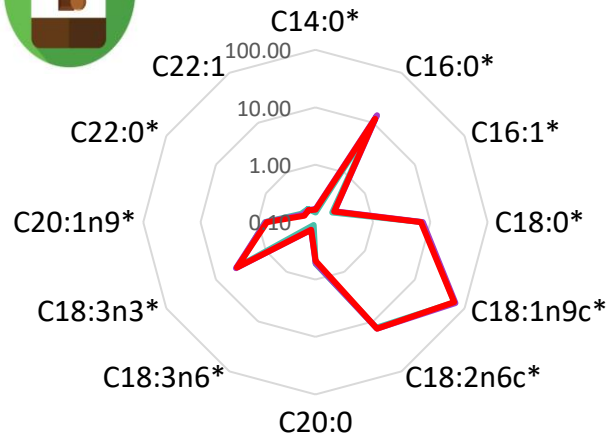






AOCS Ce2c-11

AOCS Ce2b-11



MAED

RESULTS

BF_3 VS HCl/MeOH

EXPLORE THE USE OF MICROWAVE TO PERFORM THE **DERIVATIZATION**

MAED

Microwave-assisted Extraction

EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

HEXANE EXTRACTION

TOTAL FAT

Derivatization with **BF_3**

Derivatization with **HCl/MeOH**

RESULTS

BF_3 VS HCl/MeOH

EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

DERIVATIZATION

Derivatization with **BF_3**

Derivatization with **HCl/MeOH**

MAED

BF_3 VS HCl/MeOH

EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

Derivatization with **BF_3**

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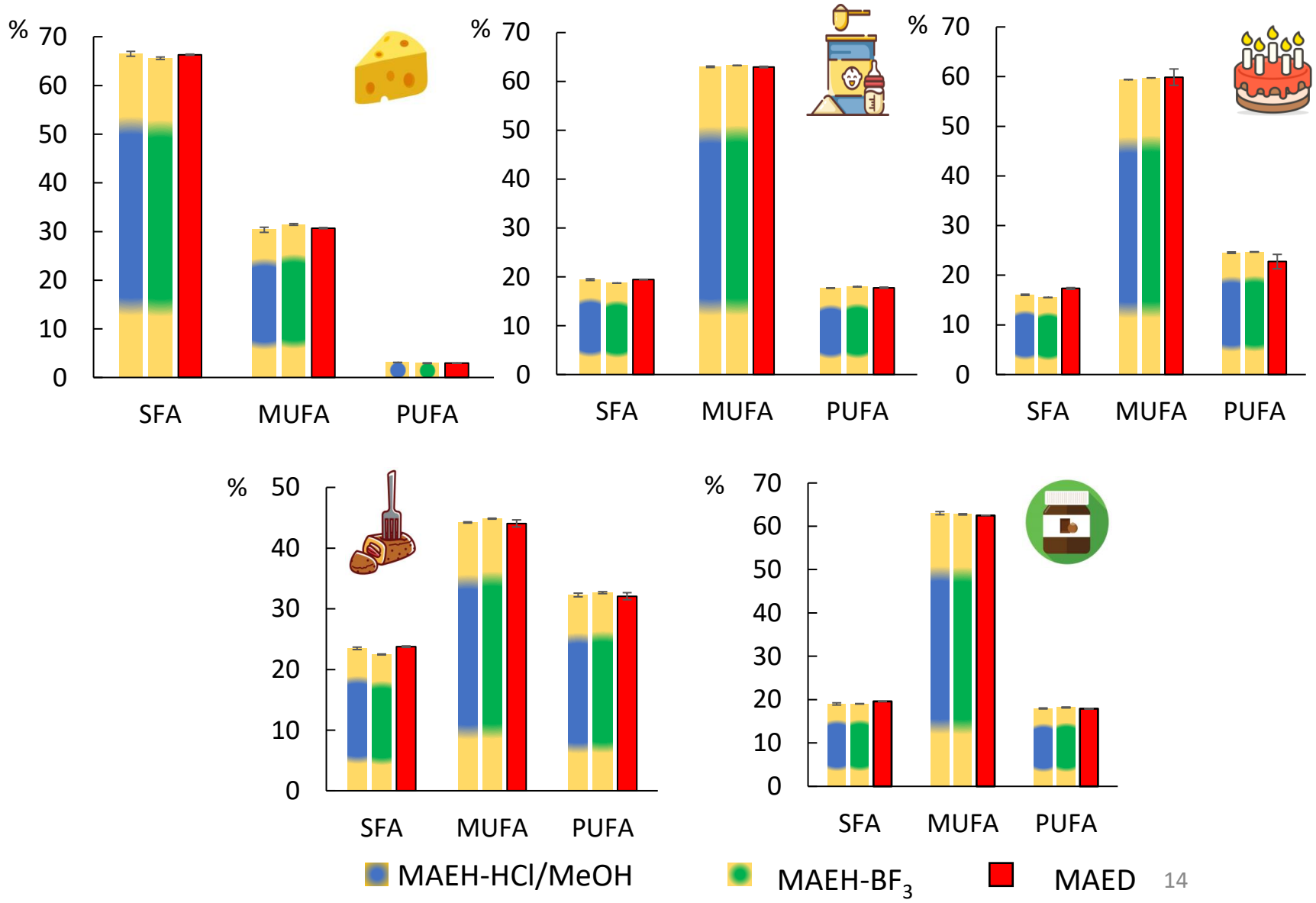
MAED



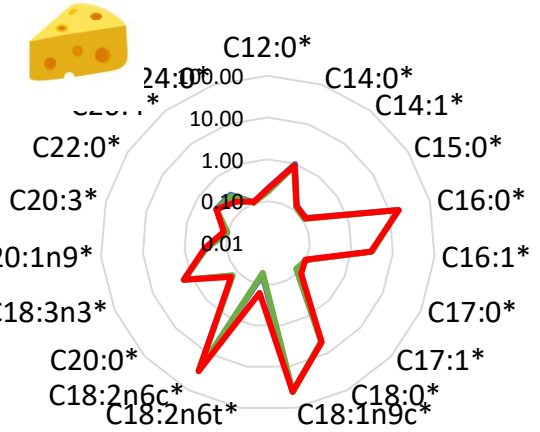
BF_3 VS HCl/MeOH

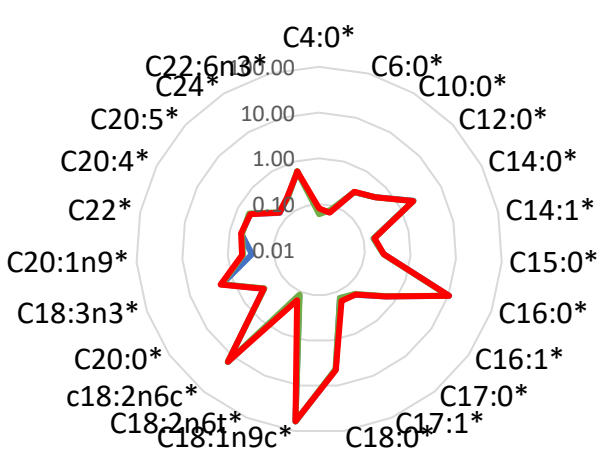
EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

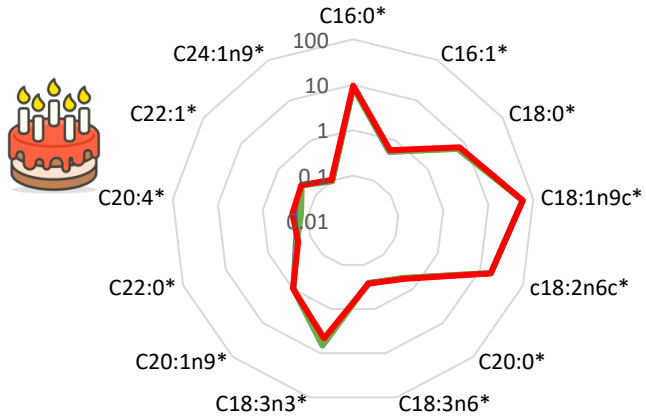
Derivatization with **BF_3**

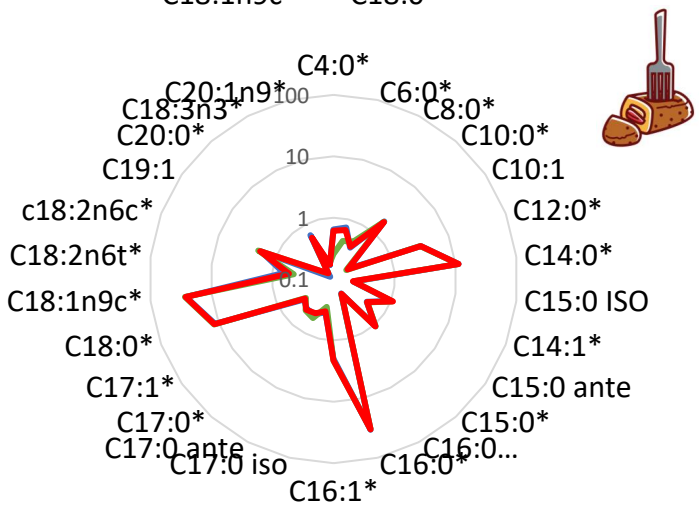
Derivatization with **HCl/MeOH**

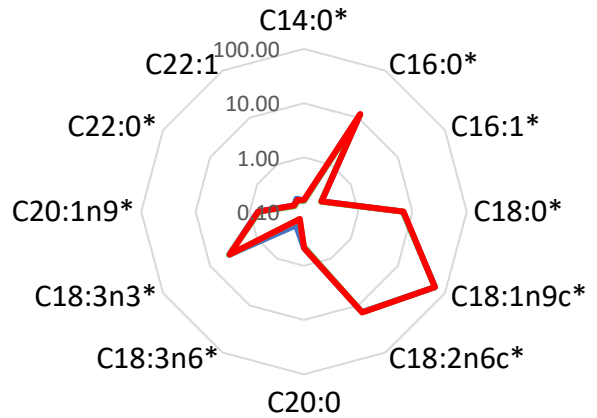
MAED











MAEH-HClMeOH MAEH-BF_3 MAED

THE SPECIAL CASE OF OAT

EXTRACTION + HYDROLYSIS
H_2SO_4 + Cyclohexane

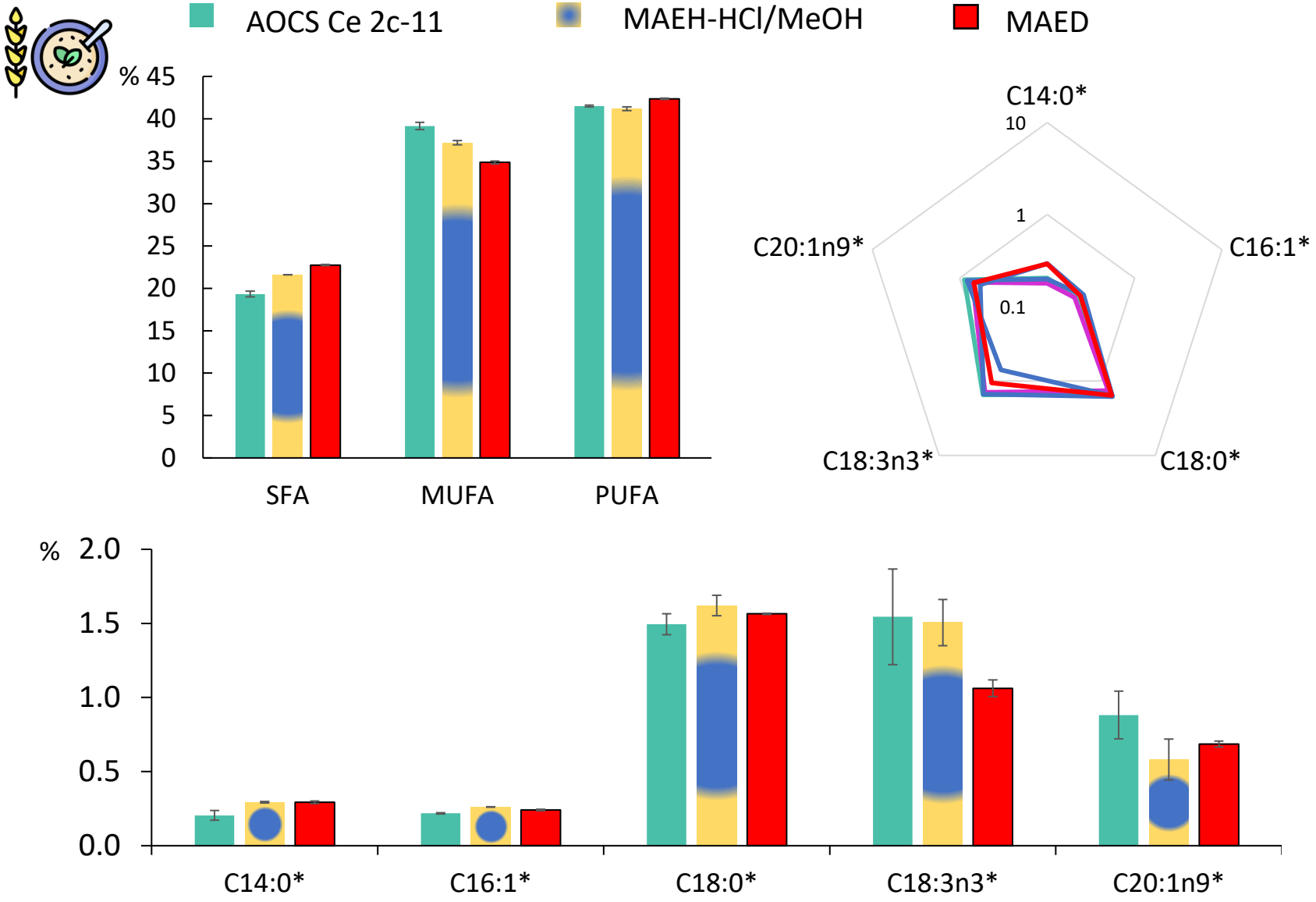
AOCS Ce2c-11

TOTAL FAT

MAED

Derivatization with
HCl/MeOH

THE SPECIAL CASE OF OAT



- the MAED one and two steps results doesn't align with the official methods
- Further studies are necessary to design an optimal MAED procedure alternative to the official method

EVALUATION OF THE GREENNESS & BLUENESS OF THE METHODS

GREENNESS

AGREEprep:
Analytical Greenness Metric for sample preparation

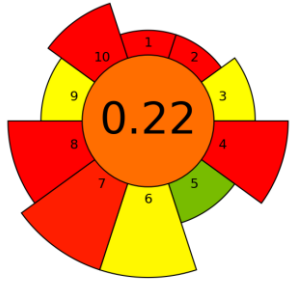
The "greenness" of a method refers to its level of environmental sustainability or "eco-friendliness." The greenness of a method is evaluated based on several criteria that measure its environmental impact. Essentially, it considers how well the method minimizes resource consumption, waste production, and the use of toxic substances.

BLUENESS

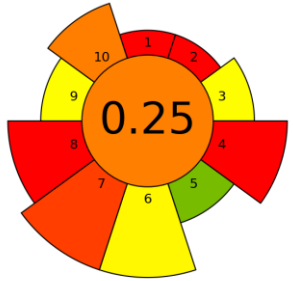
Blue Applicability Grade Index (BAGI)

The blueness of a method is referred to the practicality of the method, which is a very important parameter that is encountered by all routine analysis laboratories. BAGI can be considered complementary to the well-established green metrics, and it is mainly focused on the practical aspects of White Analytical Chemistry.

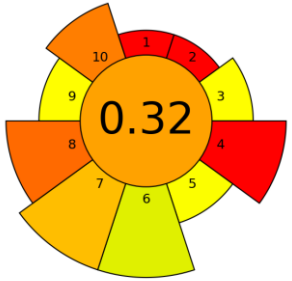
EVALUATION OF THE GREENNESS OF THE METHODS



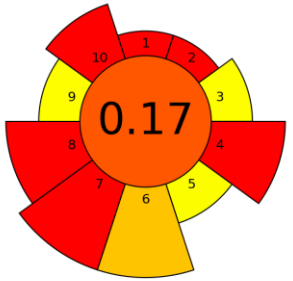
AOCS Ce 2c-11



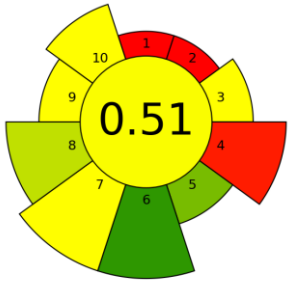
AOCS Ce 2b-11



MAEH-HCl/MeOH
MAE-HCl/MeOH

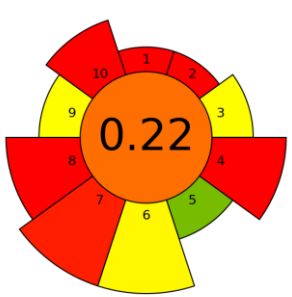


MAEH-BF_3
MAE-BF_3



MAED

EVALUATION OF THE GREENNESS OF THE METHODS



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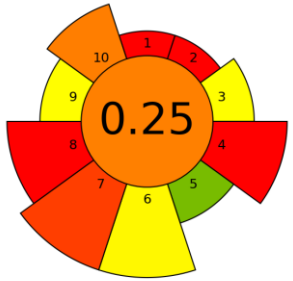
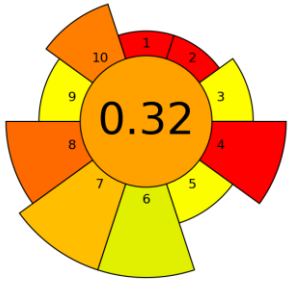
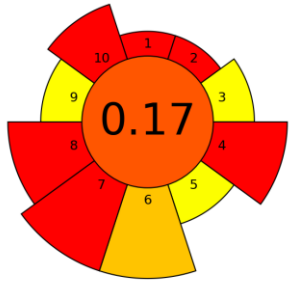


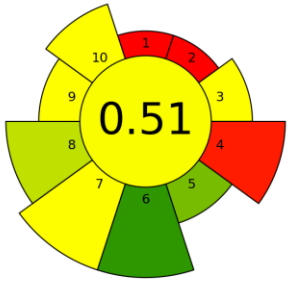
AOCS Ce 2b-11



MAEH-HCl/MeOH
MAE-HCl/MeOH



MAEH-BF_3
MAE-BF_3



MAED

HIGH THROUGHPUT

SUITABLE FOR A WIDE RANGE OF SAMPLE

AUTOMATION

LESS TOXIC SOLVENTS

IMPROVEMENT OF THE GREENNESS OF THE SAMPLE PREPARATION METHOD

EVALUATION OF THE BLUENESS OF THE METHODS

AOCS Ce 2c-11
AOCS Ce 2b-11

MAEH-HCl/MeOH
MAE-HCl/MeOH
MAEH-BF_3
MAE-BF_3

MAED

IMPROVEMENT OF THE BLUENESS OF THE SAMPLE PREPARATION METHOD

CONCLUSION

THE ONE-STEP MICROWAVE-ASSISTED EXTRACTION AND DERIVATIZATION IS COMPARABLE TO OFFICIAL METHODS AND MORE GREENER AND PRACTICAL

THE DERIVATIZATION STEP ASSISTED BY MICROWAVE CAN REPLACE THE USE OF BF_3

A SPECIAL THANKS

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THANK YOU FOR THE ATTENTION

Do you have any questions?