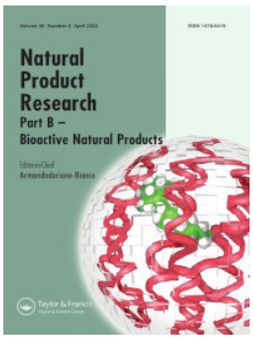

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SHORT COMMUNICATION

Microwave irradiation is a suitable method for caulerpin extraction from the green algae *Caulerpa racemosa* (Chlorophyta, Caulerpaceae)

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ABSTRACT

Caulerpin is a bisindolic alkaloid that has been obtained from many species of the genus *Caulerpa*. The main objective of this paper is to evaluate four extraction methods of caulerpin in the *C. racemosa:* maceration (DMA), Soxhlet extraction (SOX), ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE). The methods were compared through caulerpin content quantified by Ultraviolet-visible (UV-vis) spectrophotometry. The highest extract yield was obtained by SOX but the highest contain of caulerpin was presented in the MAE extract. The caulerpin content was significant different within the extacts by MAE and UAE, it yielded by MAE more than three times as much as UAE. The most efficient caulerpin extraction method had the parameters solvent, temperature and time optimised. Thus, the best conditions were achieved with MAE in ethanol during 7 min at 90 °C. Therefore, this work suggests an improved routine analysis of caulerpin by the green chemistry concept.

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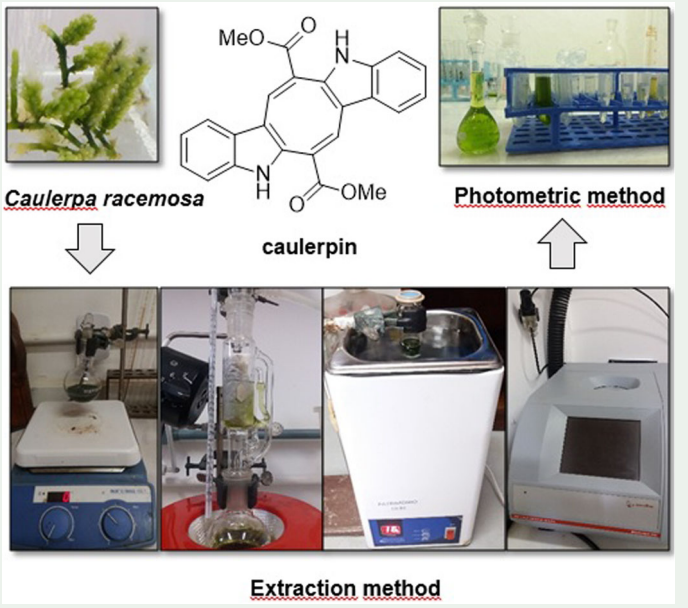
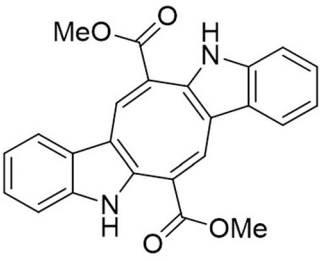


1. Introduction

Caulerpin (Figure 1) is a bisindolic alkaloid found in some species of *Caulerpa,* like *C. cylindracea*, *C. mexicana*, *C. peltata* and *C. racemosa* (Kumar et al. 2019). The common edible specie *C. racemosa* is traditionally harvested and cultivated in the Indo-Pacific (de Gaillande et al. 2017) and has caulerpin as one of the major components of its phytochemical profile (Ornano et al. 2014). Thus, there is a great interest in this seaweed due to its pharmaceutical (Senthilraja et al. 2011; Cavalcante-Silva et al. 2016) and technological applications (Kamal and Sethuraman 2012).The Green Chemistry concept (standards set by Environmental Protection Agency, USA) recommend ways to improve and optimising the extraction method of the natural source and prevent pollution by using safe solvents auxiliaries, designing energy efficiency, reducing the use of organic solvent and the time analysis (Azmir et al. 2013). In this paper, classic methods for extraction such as DMA and SOX were compared with green techniques like UAE and MAE. The quantification of caulerpin in raw material was made through UV-Vis spectrophotometry, a very simple and cheap method employed for routine analysis of commercial samples and natural products (Altemimi et al. 2017). Thus, the aim of this work was to compare four techniques and optimise the caulerpin extraction yield.

2. Results and discussion

The UV-Vis method was selective to quantify caulerpin at the maximum absorbance wavelength (λmax) 317 nm. Although the caulerpin is the major alkaloid found, there are other alkaloids recognised in *C. racemosa* extracts (supplementary information



caulerpin

Figure 1. Chemical structure of the alkaloid caulerpin.

Table S1) but no substance was reported to absorb at λmax 317 nm. The calibration curve from standard caulerpin produced a coefficient of determination (R^2) of 0.9943 and a linear regression line equation of:

$$y = 27.479x + 0.0709 \quad (1)$$

2.1. Extraction method comparison

The highest extract yield was obtained by SOX (32.062%), followed by DMA (19.502%), MAE (17.7%) and UAE (11.84%). The extracts and the isolated compound have similar spectra curves (supplementary information Figure S1). Significant difference was found in caulerpin content within the extacts by MAE and UAE (supplementary information Figure S2). The MAE contain of caulerpin was three times more than UAE. DMA and SOX showed no significant variation in caulerpin content. DMA is known to consume high quantities of solvent, have long extraction times and low extraction yields whilst SOX can reduce the solvent consumption but whit a possible degradation of thermolabile substances (Bucar et al. 2013). In this work, the MAE was 60 times faster than the SOX. Therefore, due to the advantages of MAE when compared to different methods, such as higher extraction yield over UAE, low solvent consumption when compared to DMA and faster time analysis than SOX, MAE was chosen as a suitable method for caulerpin extraction from the green algae *C. racemosa*.

2.2. Microwave extraction optimisation

In order to increase the efficiency of caulerpin extraction, we evaluated changes in the parameters solvent (methanol, ethanol and water), temperature (90, 130 and 170 °C) and extraction time (7, 21 and 35 min) of this method (supplementary information Figure S3). For the solvent parameter (supplementary information Figure S3a), ethanol presented the higher percentage of recovery. As a bio-solvent, whose have high solvent power, complete biodegradability and low toxicity, it can ensure a safe and high quality product process and reduce the generation of hazardous substances (Chemat et al. 2012). Tests on the optimisation of temperature (supplementary information Figure S3b) and extraction time (supplementary information Figure S3c) showed no significant yield variation. The MAE have the advantage of solvent reduction, level of

simplicity and automation of the operating procedures. Martinez et al. (2014) indicate it use in natural products protocols for alkaloids. Improvements on the extraction process are required to improve the development and viability of products (Chemat et al. 2012). Thus, spending less energy power is better to reduce expenses.

3. Conclusion

The extract prepared with ethanol by the MAE, at 90 °C in 7 min was the most efficient in the improved routine analysis with UV spectroscopy. It could improve the labour without lost in the caulerpin yield. Therefore, the search for sustainable, practical, shorter time, low cost and environment friendly technologies can be the new approach for the industrial use of natural products.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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