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# Infrared Irradiation, an Excellent, Alternative Green Energy Source

Joel Martínez and René Miranda

## Abstract

In this chapter, attending the request by the UNESCO, *Decade to Educate in the Sustainability*, a green-chemical approach is offered. Thus, the most recent advances using the infrared irradiation as alternative energy source are given; this is in order to activate an organic reaction in addition to conveniently perform the extractions of secondary metabolites. It is worth noting that this manuscript is an improved extent of a review previously reported by our group. The chapter is accomplished considering two sections, displaying a comprehensive overview: in a first instance, after a deep search in the literature, a broad compilation of suitable information is presented; in a second landscape, recent unpublished results from our and other laboratories are described.

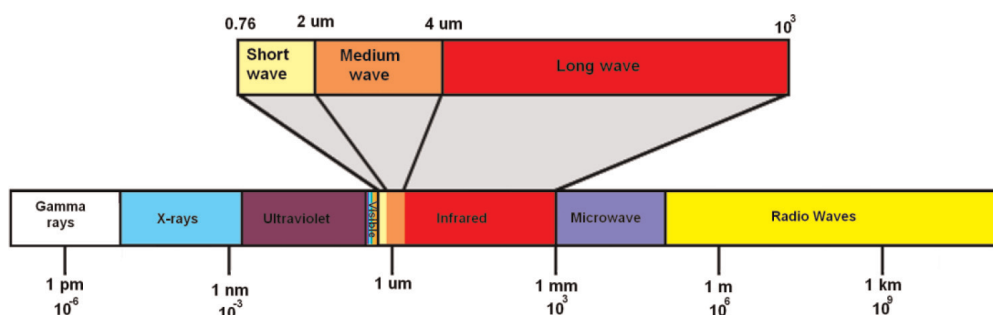
**Keywords:** infrared irradiation, green chemistry, UNESCO-DES, sustainable development, reaction activation, natural product extraction

## 1. Introduction

Since the born (early 1990s) of the paradigm of the green chemistry (GC), it has acquired its up-to-date position as a scientific discipline preventing pollution and consequently contributing to the sustainable development (SD) [1]. In this sense, it is important to keep in mind the request of the United Nations Educational, Scientific and Cultural Organization (UNESCO) performed by the establishment of the Decade to Educate in the Sustainability (DES), 2005–2014, satisfactorily evaluated in November 2014 at Aichi, Nagoya, Japan. In this sense, the UNESCO desire to improve the quality of education (teaching researching and informing) at all levels, transforming the society by the reorientation of the education adopting the sustainable development aims [2, 3].

Consequently, many educational institutions in all levels, around the world, have assumed the GC protocol as an efficient pathway to contribute within the SD using and implementing innovative technologies.

In recent years, scientists around the world have implemented strategies to avoid the use of fossil resources; in this regard, it is convenient to note that between the GC-protocol, the six principles (*energy requirements should be more recognized for their environmental and economic impacts, and they must be minimized*) [4, 5] are well recognized [6] being important to highlight that several approaches to reduce the energetic consumption have been developed in order to diminish the dependence of fossil resources: the use of microwave irradiations (MW), ultrasound (US), mechanical milling (MM), photochemical (PC), electrochemical (EC), all of them



**Figure 1.**  
*Infrared irradiation zones.*

well recognized by the GC [7]. More recently, infrared irradiation (IR) has emerged as another excellent activating source into the green chemistry efforts [1, 7].

The infrared irradiation has been already perceived by the scientific community for the convenient promotion of organic transformations, this in addition to its employment to perform efficient natural product extractions, among others process.

Regarding the last commentaries, it results appropriated to establish the goal of this chapter: provide after a profound search in the literature, the actual status concerned to the employment of the infrared irradiation as an alternative mode to activate chemical reactions in addition to demonstrate its invaluable use to perform conveniently the extraction of interesting secondary metabolites.

On the other hand, taking as supportive reference the electromagnetic spectrum, the infrared region is divided into three zones, **Figure 1**. The shortwave or near-infrared zone has band spans from 0.76 to 2 μm (NIR); the middle or medium infrared zone, with band spans from 2 to 4 μm (MIR); and the zone of long wave or far infrared with band spans from 4 to 1000 μm (FIR).

It is worth noting that the NIR offers important advantages to carry out a reaction: due to its immediate response time, because it is efficiently used by convection, in addition to the longer life time of the tungsten-halogen filament [1, 8].

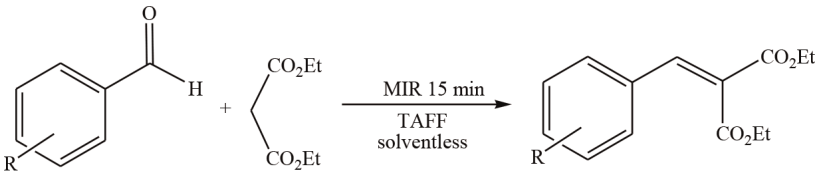
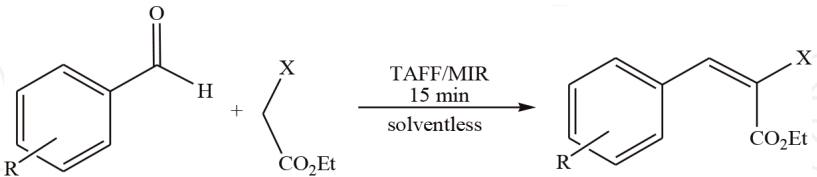
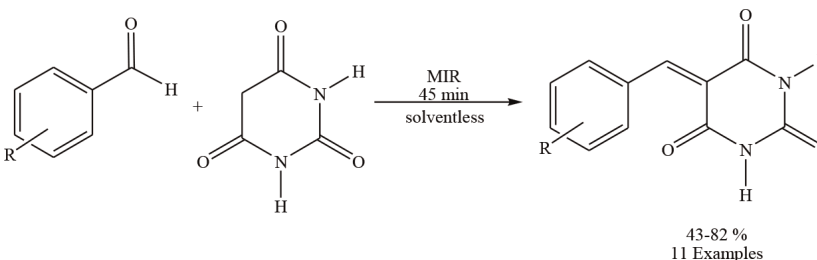
## 2. Infrared irradiation, actual use around the world

Considering the use of infrared irradiation, as an activating mode of reaction in complement to its employment to activate the extraction of secondary metabolites, in this chapter after a profound literature search, the obtained information was organized and presented in several sections. Each one of these sections was performed in order to highlight the actual status of the use of the infrared irradiation for the purposes previously commented. It is also important to note that the manuscript was created bearing in mind to attend the Decade to Educate in sustainable Development involving the green chemistry protocol.

Therefore, in the first place, the upgraded use of infrared irradiation around the world including both the early and the most recent studies in this field is showed.

### 2.1 Knoevenagel condensation

In **Table 1**, three aldol type reactions are shown, employing MIR as the activating mode, using a natural bentonitic clay—Tonsil Actisil FF (TAFF) [9] as a catalyst. The target molecules were produced with good yields, short reaction times, in the absence of solvent, and involving easiest workup.

| E | Reaction  | Ref. |
|---|---|------|
| 1 |  <p>32-73 %<br/>9 Examples</p>  | [10] |
| 2 |  <p>23-89 %<br/>27 Examples</p> | [11] |
| 3 |  <p>43-82 %<br/>11 Examples</p> | [12] |

E = Entry, Ref = Reference.

**Table 1.**  
 Knoevenagel condensation reactions.

## 2.2 Nucleophilic additions

Several nucleophilic additions to various carbonylic substrates are confined in **Table 2**; consequently different products were obtained: heterocycles, Schiff bases, and the addition of a set of sulfur derivatives or the indolyl moiety to perezone, a natural product and its synthetic analog, Entry 10; it is important to note that the obtained indolyl molecules showed good activity against breast cancer cells.

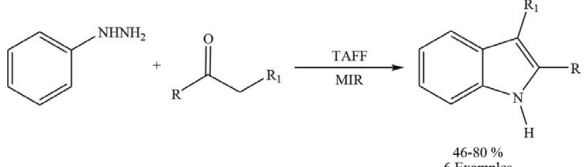
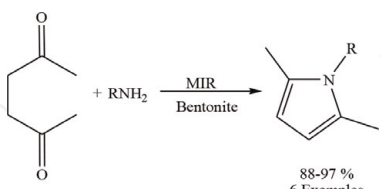
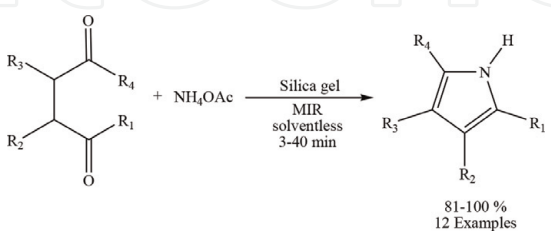
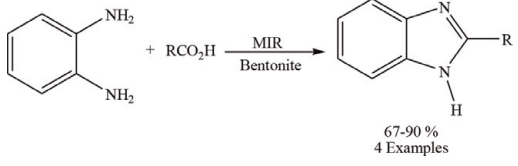
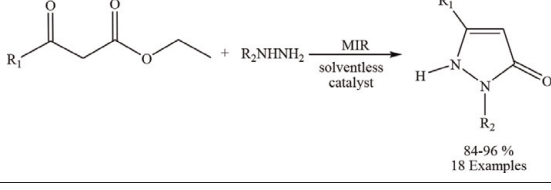
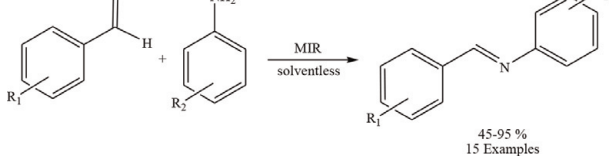
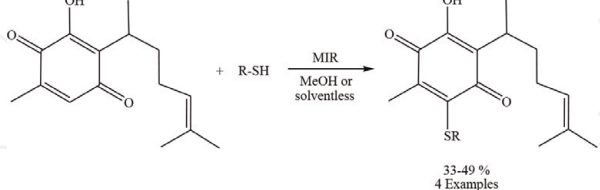
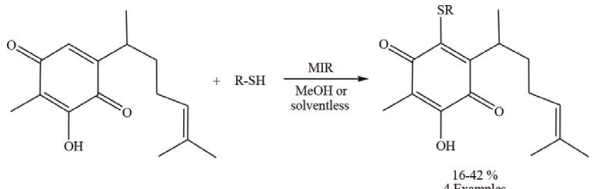
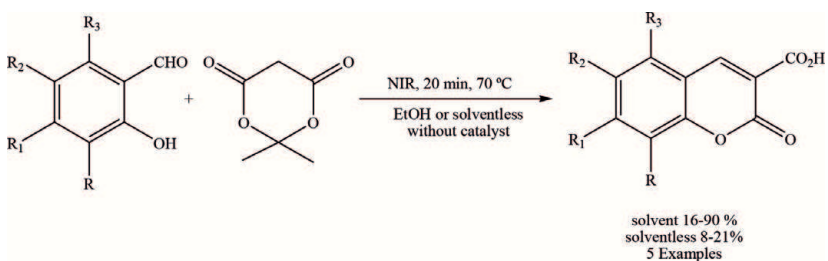
In these processes, the MIR was employed to promote the reaction; in some cases, Co doped with ZnS nanoparticles or TAFF were used as catalyst. The reactions, in general, proceed with good to excellent yields. Also, a green procedure to obtain a set of five coumarins, was developed, comparing different activating modes, MW, NIR, US, MM, and mantle heating (MH), in the presence or absence of ethanol, Entry 9.

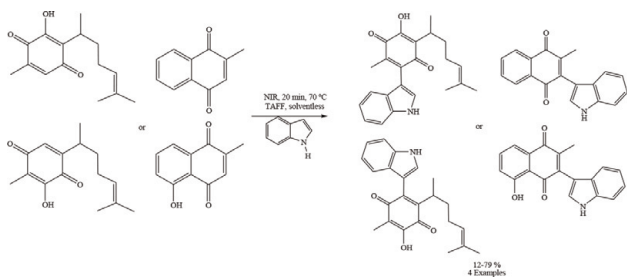
## 2.3 Related macrocycles-benzyl oligomers

Various related macrocycles or benzyl oligomers were produced in the presence of TAFF as catalyst, in absence of solvent, considering a comparative study between MIR and MW, both processes show similar yields, **Table 3**.

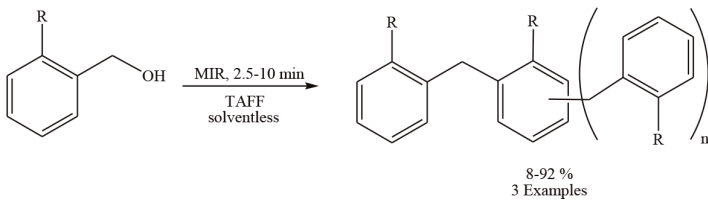
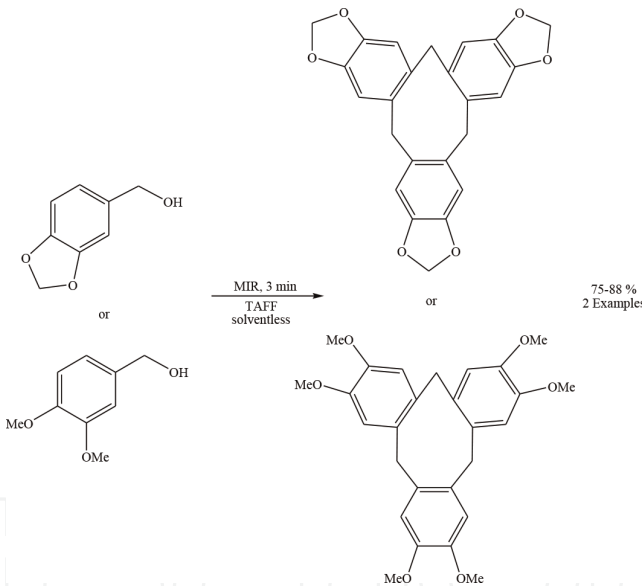
## 2.4 Multicomponent reaction

The multicomponent reaction implicates a very important and versatile strategy to generate an enormously number of reaction protocols to produce important molecules. In this sense, MIR and NIR have been usefully employed as activating reaction modes, to promote molecules related to the multicomponent protocols of

| E | Reaction   | Ref.     |
|---|--|----------|
| 1 |  <p>46-80 %<br/>6 Examples</p>                                  | [13]     |
| 2 |  <p>88-97 %<br/>6 Examples</p>                                  | [14]     |
| 3 |  <p>81-100 %<br/>12 Examples</p>                                | [15]     |
| 4 |  <p>67-90 %<br/>4 Examples</p>                                 | [16]     |
| 5 |  <p>84-96 %<br/>18 Examples</p>                               | [17]     |
| 6 |  <p>45-95 %<br/>15 Examples</p>                               | [18, 19] |
| 7 |  <p>33-49 %<br/>4 Examples</p>                                | [20]     |
| 8 |  <p>16-42 %<br/>4 Examples</p>                                | [20]     |
| 9 |  <p>solvent 16-90 %<br/>solventless 8-21%<br/>5 Examples</p> | [21]     |

| E                                  | Reaction  | Ref. |
|------------------------------------|---|------|
| 10                                 |  <p>13-79 %<br/>4 Examples</p> | [22] |
| <i>E = Entry, Ref = Reference.</i> |   |      |

**Table 2.**  
 Nucleophilic addition reaction.

| E                                  | Reaction  | Ref. |
|------------------------------------|---|------|
| 1                                  |  <p>8-92 %<br/>3 Examples</p>   | [23] |
| 2                                  |  <p>75-88 %<br/>2 Examples</p> | [23] |
| <i>E = Entry, Ref = Reference.</i> |   |      |

**Table 3.**  
 Formation of related benzylic oligomers and macrocycles.

Biginelli, Hantzsch Meldrum, and Kamal-Qureshi (**Table 4**). The target molecules were in general with convenient green approach: without solvent or the use of EtOH as solvent, employing a green catalyst. The reported procedures involved short reaction times and easy workup.

## 2.5 Redox procedures

Important practices, within organic chemistry, are the reduction-oxidation, procedures. Thus, into the green chemistry protocol, several attempts have been developed, particularly within photocatalysis field, for example Guo and coworkers present the photothermic reduction of graphene oxide employing MIR [38].

| E | Reaction | Ref. |
|---|----------|------|
| 1 |          | [24] |
| 2 |          | [24] |
| 3 |          | [25] |
| 4 |          | [26] |
| 5 |          | [26] |
| 6 |          | [27] |
| 7 |          | [27] |
| 8 |          | [28] |



| E  | Reaction                       | Ref.     |
|----|--------------------------------|----------|
| 9  | <p>50-75 %<br/>10 Examples</p> | [29]     |
| 10 | <p>59-65 %<br/>2 Examples</p>  | [29]     |
| 11 | <p>55-96 %<br/>9 Examples</p>  | [30]     |
| 12 | <p>40-83 %<br/>12 Examples</p> | [31]     |
| 13 | <p>40-98 %<br/>12 Examples</p> | [32]     |
| 14 | <p>65-85 %<br/>14 Examples</p> | [33]     |
| 15 | <p>12-67 %<br/>15 Examples</p> | [34]     |
| 16 | <p>54-95 %<br/>20 Examples</p> | [35, 36] |



| E  | Reaction | Ref. |
|----|----------|------|
| 17 |          | [37] |

E = Entry, Ref = Reference.

**Table 4.**  
Multicomponent reaction.

Another example, using NIR, is the photocatalytic decomposition of 2,4-dichlorophenol in aqueous solution with  $\text{Cu}_2(\text{OH})\text{PO}_4$  microcrystals [39], or the photo-oxidation of 1,3-diphenylisobenzofuran in aerated toluene [40]. Other oxidation processes are presented in **Table 5**, developed without solvent, excellent yields, and short time reaction.

## 2.6 Miscellaneous reactions

An interesting set of miscellaneous reactions is commented in this separated section. In this sense, the production of peptides by mean *N*-phosphoamino acid and amino acid in aqueous solution and irradiated with MIR was reported [43]. Also, the alpha-alumina powders were described by mean Pechini synthesis using MIR [44]. In other studies,  $\text{SnO}_2$  nanoparticles were generated applying medium infrared irradiation [45]. The hexagonal form of boron nitride was employed to produce protective coatings using liquid polyborazylene as a boron source and was activated by MIR [46].

In a current research, the preparation of botanical samples enriched with organic matter by mean heating of focused far infrared light by dry ashing method is reported. In the dry process, the NIR was focused by reflection through the gold-plated layer; consequently, the energy efficiency was improved and a argon atmosphere was employed to avoid the loss of elements caused by sample combustion at high temperatures,  $500^\circ\text{C}$ , in short times of carbonization. It is important to note that this device shows several advantages as the instrument size is small compared

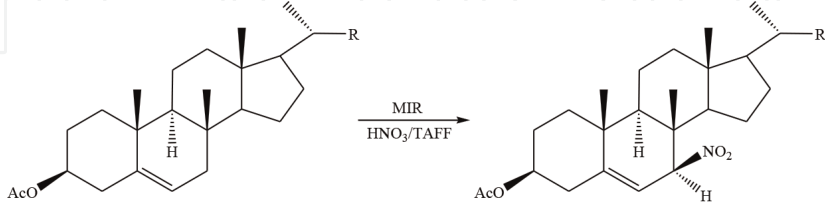
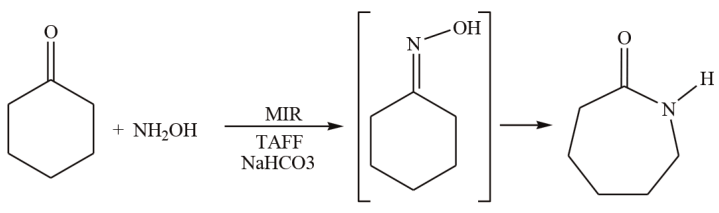
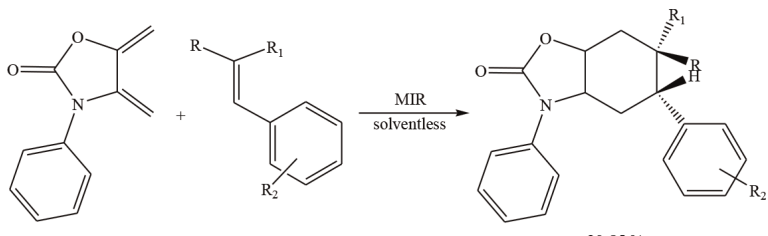
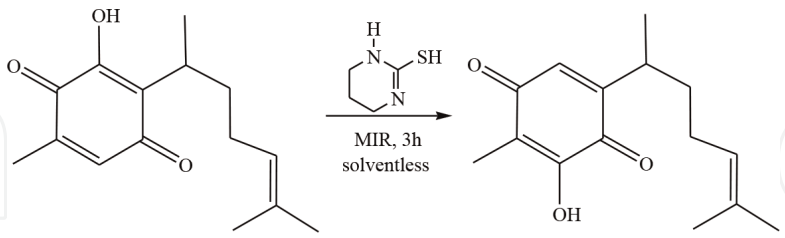
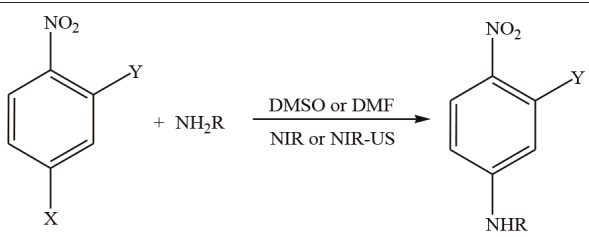
| E | Reaction | Ref. |
|---|----------|------|
| 1 |          | [41] |
| 2 |          | [42] |

E = Entry, Ref = Reference.

**Table 5.**  
Reduction-oxidation reactions.

with electric muffle furnace and has a fast heating rate the gold-plates improve the energetic efficiency; and in general, the ashing process is completed within 30 min, among others [47].

As a complement, in **Table 6**, several reactions are summarized as described in a previous report [1]. The included processes, in general involve short reaction times, good to excellent yields, the use of a catalyst and solventless conditions, and consequently a green approach is offered.

| E | Reaction  | Ref. |
|---|---|------|
| 1 |  <p>14-18 %<br/>2 Examples</p>                        | [48] |
| 2 |  <p>92 %<br/>1 Example</p>                           | [49] |
| 3 |  <p>20-85 %<br/>10 Examples</p>                     | [33] |
| 4 |  <p>45 %<br/>1 Example</p>                          | [20] |
| 5 |  <p>NIR 9-93 %<br/>NIR-US 6-77 %<br/>32 Examples</p> | [50] |

E = Entry, Ref = Reference.

**Table 6.**  
 Miscellaneous reactions.

Recently, several nucleophilic aromatic substitutions have been reported by Luna-Mora and coworkers; the authors inform about the preparation of *N*-(5-*R*<sup>1</sup>amino-2-nitrophenyl) acetamides and 5-*R*<sup>1</sup>amino-2-nitroanilines by means of a comparative study among MH, NIR, US, MW, and the combination of NIR-US here is concluded that the best process was when the NIR-US process is employed offering the best yields and short reaction times, Entry 5.

## 2.7 C–C bond formation

The C–C bond formation is a versatile tool to generate various compounds; some examples are presented in **Table 7**, the products were generated in good to excellent

| E | Reaction                       | Ref. |
|---|--------------------------------|------|
| 1 | <p>30-98 %<br/>7 Examples</p>  | [37] |
| 2 | <p>91-99 %<br/>10 Examples</p> | [51] |
| 3 | <p>45-97 %<br/>10 Examples</p> | [52] |
| 4 | <p>65-94 %<br/>20 Examples</p> | [53] |
| 5 | <p>55-99 %<br/>16 Examples</p> | [53] |
| 6 | <p>65-99 %<br/>15 Examples</p> | [54] |

*E* = Entry, *Ref* = Reference.

**Table 7.**  
Formation of C–C bond.

yields, using NIR as the activating mode of reaction, with short reaction times in the presence of a catalyst, with or without the presence of a base.

Related to **Table 7**, several interesting commentaries are performed:

Balam-Villareal in 2016 informed about the formation of C-C bond by means of a Mizoroki-Heck cross-coupling reaction, Entry 3, employing new sulfur-containing palladacycles as catalyst and aryl iodides with electron-donating and electron-withdrawing groups. Thus the NIR strategy, has similar behavior that the MH in relation to the yields obtained, but the time reaction was diminished by three times.

Another example, corresponding to C-C bond formation, implicates the use of arylhydrazones containing the benzothiazole moiety as a contribution to the Mizoroki-Heck reaction, employing palladium ligands, Entries 4 and 5. The authors report that the reaction was performed using near infrared irradiation. The products were obtained from good to excellent yields, reducing the reaction time. In the same report, it was reported the use of this arylhydrazones in the Suzuki-Miyaura cross-coupling reaction with NIR as alternative source, water as solvent, diminishing the time of reaction, and the products were obtained with good yields.

Continuing with the Suzuki-Miyaura reaction type, the use of imidazole-hydrazone as ligand was recently informed; the reaction was developed in short reaction times, improving the yields, all this result afforded by the use of NIR as the activating mode of reaction, with water as the solvent, offering a new ecological alternative to perform this kind of reaction, Entry 6.

2.8 Natural product extraction

Currently, it is well known that several methods are available to extract the secondary metabolites present in vegetable species, but recently, it was reported that the FIR can be also used [1] (**Table 8**).

| E  | Vegetable specimen                            | Extracted compounds  | Ref.     |
|----|---|--|----------|
| 1  | <i>Oryza sativa</i> L.                        | <i>p</i> -Coumaric acid, 3-vinyl-1-oxybenzene, 4-hydroxybenzaldehyde, among others   | [55, 56] |
| 2  | <i>Sesamum indicum</i> L.                     | <i>p</i> -Hydroxybenzoic acid, <i>o</i> -coumaric acid, vanillic acid, <i>p</i> -coumaric acid, isofeluric acid, sesamol, and tocopherol | [57]     |
| 3  | <i>Arachis hypogaea</i> L.                    | 2-Methoxyphenol, 2-hydroxy-4-methoxybenzoic acid, among others   | [58]     |
| 4  | <i>Camellia sinensis</i> var. <i>sinensis</i> | Polyphenolic compounds, caffeine, (–)-epicatechin, among others  | [59–61]  |
| 5  | <i>Puerariae radix</i>                        | Polyphenols compounds, isoflavonoids compounds   | [62]     |
| 6  | <i>Morus alba</i> L.                          | <i>p</i> -Coumaric acid, benzoic acid, (+)-catechin, chlorogenic acid, among others  | [63]     |
| 7  | <i>Radix Salviae miltiorrhizae</i>            | Danshensu, protocatechuic acid, protocatechuic aldehyde, among others  | [64]     |
| 8  | <i>Lycium barbarum</i> Linn                   | Quercetin, rutin, and gentisic acid  | [65]     |
| 9  | <i>Glycyrrhiza uralensis</i> Fisch            | Liquiritin, glycyrrhetic acid, and glycyrrhizin  | [66]     |
| 10 | <i>Lycium chinensis</i> Mill                  | Mannitol, sucrose, glucose, and fructose   | [67]     |
| 11 | Grape seeds                                   | Catechin, epicatechin, and procyanidin B2  | [68]     |
| 12 | <i>Rhododendron mucronulatum</i> Turcz        | Rutin, farrerol, syringic acid, vanillic acid, and 4-hydroxybenzoic acid   | [69]     |

| E  | Vegetable specimen                          | Extracted compounds  | Ref. |
|----|---|--|------|
| 13 | <i>Tagetes erecta</i> L.                    | <i>p</i> -Coumaric acid, gallic acid, among others                 | [70] |
| 14 | <i>Picrorhiza scrophulariiflora</i> Pennell | Picroside I and picroside II                                       | [71] |
| 15 | <i>Hibiscus cannabinus</i> L.               | Polyphenolic and flavonoids compounds, kaempferitrin, among others | [72] |
| 16 | <i>Glycine max</i> M.                       | Daidzein, and genistein  | [73] |
| 17 | <i>Fagopyrum</i> spp.                       | Polyphenols and flavonoids   | [74] |
| 18 | Apricot                                     | Polyphenolic, flavonoids, and tannins compounds                    | [75] |
| 19 | Habanero pepper                             | Capsaicin and dihydrocapsaicin                                     | [76] |

*E* = Entry, *Ref* = Reference.

**Table 8.**  
*Extraction of natural compounds.*

Interesting and recent information corresponds to a study by Cheaib and coworkers, Entry 18, reporting the extraction comparing the solid/liquid extraction with infrared-assisted extraction. In this study, the FIR pomace extract, from apricot, gave the highest polyphenolic content, flavonoid, and tannin yields. In addition, the inhibitory activity study against gram-positive and gram-negative bacteria was performed, and the infrared pomace gave the highest activity in comparison with infrared kernel, solid-liquid pomace, and solid-liquid kernel.

More recently, Entry 19, Martínez and coworkers have reported the extraction of capsaicin and dihydrocapsaicin, from habanero pepper in a comparative study, the use of NIR as source of extraction with EtOH as solvent, showed the best yields, 43.88 and 29.44% for capsaicin, and dihydrocapsaicin, respectively. The effect of nonconventional energies was analyzed by means of SEM micrographs, resulting in an NIR-procedure with a higher number of intact particles in the cellular matrix, in comparison with nonirradiated material.

**3. Unpublished results from our and other laboratories**

The results showed in this section correspond to novel results, being important to note that they have been recently submitted for publication or are under writing.

A green contribution to produce SiO<sub>2</sub>–TiO<sub>2</sub> catalyst is offered, using NIR in comparison with other nonconventional energy sources and mantle heating. According to the obtained results, the materials were produced in short time reaction employing water as solvent. The results, for NIR strategy, show that smaller crystal size (15.3 nm) and higher BET surface area were obtained (335.2 m<sup>2</sup>/g). In addition, the surface area obtained by mean NIR procedure, in comparison with other process, is due to that the water in the reaction was partially evaporated, affecting the hydrolysis process, but the pore and volume diameter was improved (4.33 nm and 0.363 cm<sup>3</sup>/g, respectively) in comparison with the MH mode(2.77 nm and 0.290 cm<sup>3</sup>/g) [77].

Related to natural product extraction, Miranda et al. have achieved a wide study about the extraction of perezone, from roots of both *Acourtia adnata* and *Acourtia platyphylla* comparing various modes: MH, NIR, MW, US, and supercritical CO<sub>2</sub>. It is appropriated to comment that the yield obtained from the extraction with

NIR for 15 minutes was statically equal to the conventional thermal extraction, reflux during 3 h [78, 79].

Penieres-Carrillo and coworkers currently have developed the acylation of aromatic amines and alcohols, by a comparative study among MH, US, IR, and IR-US, with preliminary results; the best process has been the simultaneous use of IR-US, with high yields and shorter reaction times [80].

#### **4. Conclusions**

The request, “Decade to Educate in the Sustainability,” established by the UNESCO (2005–2014) satisfactorily evaluated in November 2014, at Aichi, Nagoya, Japan is attended in this chapter, in this sense by means of a green approach using the infrared irradiation as an alternative and efficient mode to activate an organic reaction in addition to conveniently performed extractions of secondary metabolites. Moreover, it is important to note that this green energy can be used in other areas as material engineering and other synthetic strategies as Suzuki-Miyaura or Mizoroki-Heck cross-coupling can use it to activate the reaction. Consequently, the infrared irradiation can extend their application to other organic transformations or disciplines.

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#### **Conflict of interest**

The authors declare no conflict of interest.

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