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Microwave assisted green synthesis of aryl methoxylated benzamides and 2-oxazolines from biomass

Abstract: This paper presents a simple, rapid and efficient green synthesis of aryl methoxylated benzamides and 2-oxazolines from renewable *Eucalyptus* biomass – tar derivatives. One-pot synthesis was realized using microwave irradiation as an alternative energy source, through a direct condensation reaction of amino alcohol with carboxylic acids as starting materials. In comparison to classical organic synthesis, the protocol proposed in this work offers advantages, such as the use of common and low cost starting materials, simple work up, solvent-free reaction, shorter reaction time, good chemical conversion process and absence of catalyst.

Keywords: 2-aryl-2-oxazolines; benzamides; microwave irradiation; renewable biomass; wood tar.

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1 Introduction

Eucalyptus species are widely used in the reforestation process in Brazil and other countries. The wood of *Eucalyptus* sp. has been submitted to a rapid carbonization for charcoal production. In steel industries, vegetable charcoal is used as a thermo reducing agent, to convert iron ore to steel. However, the carbonization of wood results in low charcoal yields, approximately 30%, and the production of large amounts of different volatiles substances, which are released into the atmosphere, constituting an environmental problem. In order to minimize this environmental hazard and bring aggregate values to the charcoal production, some steel industries recover the volatiles by

a condensation process. The liquid mixture resulting from volatiles condensation is named pyrolygneous liquor, which after decantation, produces an aqueous phase (pyrolygneous acid) and an organic phase (*Eucalyptus* tar). *Eucalyptus* tar is a complex mixture, rich in phenolic and carbonylic compounds, formed during the thermal decomposition of the wood components [1]. Thus, the use of *Eucalyptus* tar derivatives from renewable biomass as starting materials, in the substitution of petrochemical derivatives, represents an adequate option in terms of green organic synthesis.

The great interest in aryl methoxylated compounds is related to their biological activities. For example, trimethoprim is a bioactive component in the formulation of commercial antibacterial drugs found worldwide [2]. Recently, bornyl benzoates were synthesized and showed activity against *Trypanosoma cruzi* [3]. In this case, the association of the properties of terpenes with aryl methoxylated compounds, to produce bioactive esters, has been successful. By this process, several compounds with potential biological activity can be synthesized from aryl methoxylated carboxylic acids, such as amides and 2-oxazolines.

Some properties have dictated the use of 2-oxazolines as monomers in polymer production, as moderators in analytical processes, and as conformational rigid peptide mimics in medicinal chemistry. Substituted 2-oxazolines have been investigated widely for pharmaceutical uses [4–6]. Especially useful as tranquilizing agents and central nervous system regulators are the substituted 2-amino-2-oxazolines. Much use has been made of the chemistry of oxazolines and their correlation with chloramphenicol for the synthesis of that antibiotic [4–6].

The amide function is found in many bioactive compounds, therefore is important to understanding its formation, properties and reactions, especially those related to the chemistry of proteins and polypeptides [7]. In addition, amides are important intermediates in the synthesis of other classes of compounds, such as amines, nitriles and oxazolines [8].

The 2-oxazolines are five-member heterocyclic compounds constituting a simple cyclic imino ester, which is found in nature as part of the structure of macromolecules

of some marine organisms. Substances containing 2-oxazoline have been isolated and their attractive biological activity, as well as their role as chelating agents, has inspired synthetic and structural studies [9].

In organic synthesis, 2-oxazoline has demonstrated a vast synthetic potential as the protected form of hydroxy-amino acids and amino alcohols, remarkably efficient director for *O*-lithiation of benzene ring and nucleophilic aromatic substitution [4–6]. Additionally, chiral oxazolines represent versatile building blocks for the synthesis of polyfunctionalized compounds, and are widely used as chiral ligands or chiral auxiliaries for regio- and stereochemical control in asymmetric synthesis [10, 11].

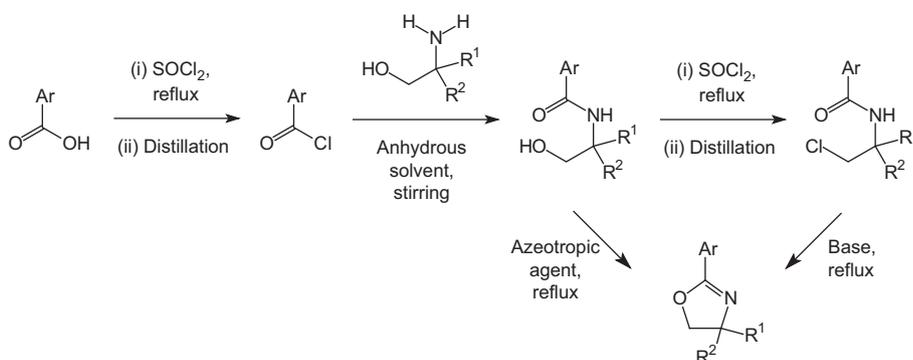
The 2-oxazolines can be synthesized by means of different routes [4–6]. A traditional method employs the conversion of carboxylic acid in acyl chloride derivative, followed by its reaction with the appropriate amino alcohol, under cyclodehydration conditions (Scheme 1). However, the incomplete reaction, harsh conditions and a mixture of products formed by this kind of reaction, have limited its use. The cyclization of the hydroxyamide intermediate induced by thionyl chloride, in the presence of a base, is an alternative route (Scheme 1). Nevertheless, this procedure also presents some limitations associated with the high reactivity of thionyl chloride, and its decomposition which leads to *in situ* formation of hydrogen chloride, which can induce undesirable side reactions, such as demethylation, hydrolysis and/or decarboxylation. As a result, the formation of decomposition byproducts mixtures decreases the yield of 2-oxazoline. Different alternative methodologies have been developed in order to avoid the use of thionyl chloride, to induce ring closure of the amide intermediate into 2-oxazoline. Some limitations were observed in the related synthesis, such as the use of multistep and complex reactions, the presence of specific reagents and unusual conditions [12].

Commercially, great interest has been focused on derivatives of 2-oxazolines, due to low cost, like carboxylic acids and amino alcohols. To reach this objective, the amino alcohols should present in their structure, a primary amino group and a hydroxyl group on adjacent carbon atoms. In addition, the most reactive β -amino alcohols are those having complete substitution on the carbon atom on which the amino group is attached. However, the direct synthesis of 2-oxazoline by the cyclodehydration reaction between carboxylic acid and amino alcohol, requires a high temperature, an azeotropic or other dehydrative agent, and a very long time reaction [4–6].

The availability of new processes and technologies in green chemistry [13] has aroused the attention of researchers, for the conversion of renewable biomass in fine chemicals for several segments, such as pharmaceuticals, food additives, materials and many others. Methoxylated benzamides and 2-aryl-2-oxazolines are important intermediates in the synthesis of natural products which possess biological and/or medicinal activity, such as hasubanan alkaloid analogues [14]. In addition, these compounds are valuable intermediates for the preparation of several compounds, such as unusual amino acids [10] and tetrazoles [15], for example, the losartan analogues [16].

In this sense, the application of microwave (MW) irradiation as alternative energy source in organic synthesis has become an increasingly widespread practice in research works and also in industry, because it can promote a rapid and selective heating of materials, as well as providing clean chemical processes, both associated with low cost [17].

There are some examples of MW-assisted synthesis of 2-oxazolines. Crosignani et al. reported the cyclization of *N*-(β -hydroxy)amides, promoted by *N,N*-diisopylcarbodiimide (DIC) and Lewis-acid catalysis (5 mol% $\text{Cu}(\text{OTf})_2$), to give the corresponding 2-oxazolines [18]. Sharma et al., recently reported the synthesis of 2-oxazolines from direct



Scheme 1 Traditional routes for preparation of 2-oxazolines.

condensation of carboxylic acids, with excess 2-amino-2-methyl-1-propanol, in good yields [19].

Combining the advantages of MW irradiation and the use of a biomass derivative, this work aims for the synthesis of aryl methoxylated benzamides and 2-oxazolines from a direct reaction between amino alcohols with carboxylic acids obtained from *Eucalyptus* tar. Thus, the *Eucalyptus* tar derivatives 3,4,5-trimethoxybenzoic acid (**1**) and 3,4-dimethoxybenzoic acid (**2**) (veratric acid) were used as starting materials in the synthesis of aryl methoxylated benzamides (**3a,b**) and 2-oxazolines (**4a,b**) (Scheme 2).

2 Experimental

Carboxylic acids **1** and **2** were obtained from chemical transformations of *Eucalyptus* tar constituents [20]. 2-Amino-2-methyl-1-propanol (Sigma-Aldrich, Seelze, Lower Saxony, Germany) was purified by reduced pressure distillation and dried under anhydrous sodium sulfate (Synth, Diadema, Sao Paulo, Brazil) before use. Flash column chromatography [hexane/ethyl acetate (Sigma-Aldrich, Seelze, Lower Saxony, Germany)] was carried out with silica gel 60G (Merck Chemicals Ltd, Nottingham, England, UK) (40–63 μm). All other chemicals were of analytical grade and used as received.

A domestic MW oven (Electrolux, Industrial District-Manaus, Amazonas, Brazil) (ME21S, 1300 W), adapted with safety devices (cooler, reflux condenser and water circulation tube) for chemical reactions, was used for the synthesis reactions. The control of the power applied and the reaction time allowed adequate conditions for preferential formation of benzamides and 2-oxazolines to be established.

The melting points reported were not corrected. Infrared analyses were performed using a Shimadzu/IR-408 spectrophotometer (Shimadzu, Tokyo, Japan). ^1H and ^{13}C NMR spectra were recorded on Bruker Avance DRX200

(Bruker, Billerica, MA, USA), using TMS as the internal standard ($\delta_{\text{H}}=\delta_{\text{C}}=0$). GC-MS analyses were performed using Agilent Technologies CG 7890/MSD 5975 (Agilent Technologies, Santa Clara, CA, USA). Instrument control parameters were: column HP-5MS, 30 m, 0.32 mm i.d., 0.25 μm film thickness, He as carrier gas. Oven program: equilibration time 0.5 min, 140°C for 1 min, then 10°C min^{-1} to 280°C for 5 min; run time 20 min.

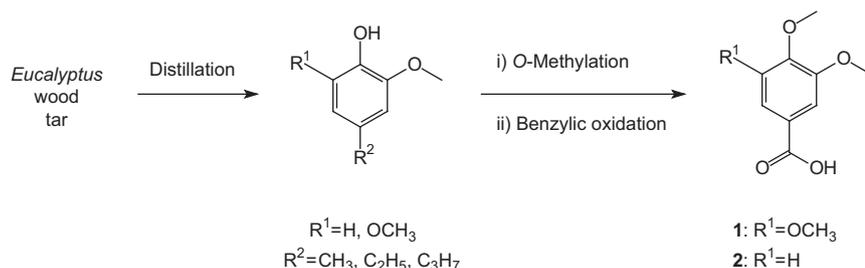
The following procedure illustrates the general method used for preparation of benzamides (**3a,b**) and 2-aryl-2-oxazolines (**4a,b**). A mixture of carboxylic acid and amino alcohol was added in a distillation balloon (50 ml) coupled to a condenser and the mixture was submitted to MW irradiation in different experimental conditions, according to the reactivity of starting materials (Table 1).

The crude products were purified by liquid-liquid extractions, followed by column chromatography. The organic layers from the extractive processes were dried with anhydrous sodium sulfate. A rotary evaporator (Tecnal, Piracicaba, Sao Paulo, Brazil) was used to concentrate the dried extracts. The reactions were monitored by TLC and GC-MS. Solvents and reagents were recovered from the purification steps and reused.

Hydroxy amides (**3a,b**) and 2-aryl-oxazolines (**4a,b**) derived from 2-amino-2-methyl-1-propanol are known substances and the chemical structures were identified by comparison with physical and spectral (FTIR, NMR and GC-MS) data [21, 22].

The side-products formed are known compounds (**5, 6**) and chemical structures were identified by comparison of the GC-MS profiles with spectral data of the NIST MS Search 2.0 Library.

N-(2-hydroxy-1,1-dimethyl)-3,4,5-trimethoxybenzamide (3a): white solid, mp 101–103°C, $\text{C}_{14}\text{H}_{21}\text{NO}_5$, MW=283.32 g mol^{-1} . **FTIR** (KBr): 3410, 3290, 3090, 3000, 2990, 2960, 2890, 2850, 1640, 1590, 1560, 1500, 1475, 1420, 1350, 1240, 1175, 1125, 1075, 1000, 970, 850, 775, 740, 725, 670, 600, 590. **MS** (EI, 70 eV) m/z (%): M^+ . 283 (6), 252 (7), 250 (5), 212 (12), 195 (100), 167 (3) 152 (3), 124 (1). **^1H NMR**



Scheme 2 Preparation of carboxylic acids **1** and **2** from biomass.

Table 1 Reaction conditions for preparation of amides (**3a**, **3b**) and oxazolines (**4a**, **4b**).

Experimental conditions	Protocol			
	A	B	C	D
Carboxylic acid	1	1	2	2
	0.86 mmol	0.86 mmol	1.00 mmol	1.00 mmol
2-Amino-2-methyl-1-propanol	5.00 mmol	5.00 mmol	2.00 mmol	2.00 mmol
Total power (kW)	5.27×10^2	6.24×10^2	4.57×10^2	6.24×10^2
Reaction time (min)	8	12	8	8
GC conversion (%)	94,75 (3a)	36,35 (4a)	31,78 (3b) 55,46 (4b)	100,00 (4b)

(200 MHz, CDCl_3), δ (ppm): 6.94 [Ar-H, s, 2H], 6.08 [NH, 1H], 3.91 [OCH_3 , s, 6H], 3.88 [$-\text{OCH}_3$, s, 3H], 3.70 [OCH_2 , s, 2H], 1.43 [CH_3 , s, 6H]. $^{13}\text{C NMR}$ (50 MHz, CDCl_3), δ (ppm): 168.08 [C=O], 153.22 [C (C-3,5)], 141.19 [C (C-4)], 130.24 [C (C-1)], 104.47 [CH (C-2,6)], 70.69 [CH_2], 60.92 [CH_3], 56.51 [C], 56.42 [C], 51.41 [C], 24.73 [CH_3].

N-(2-hydroxy-1,1-dimethyl)-3,4-dimethoxybenzamide (3b): yellow oil, $\text{C}_{13}\text{H}_{19}\text{NO}_4$, MW=233.30 g mol $^{-1}$. **MS** (EI, 70 eV) m/z (%): M^+ . 253 (2), 222 (8), 182 (11), 165 (100), 137 (8), 122 (5), 94 (5), 92 (5), 77 (11), 51 (5), 44 (16). $^1\text{H NMR}$ (200 MHz, CDCl_3), δ (ppm): 7.25 [Ar-H (H-2), d ($J=2.0$ Hz), 1H], 7.17 [Ar-H (H-6), dd ($J=8.3$ Hz, $J=2.0$ Hz), 1H], 6.67 [Ar-H (H-5), d ($J=8.3$ Hz), 1H], 6.64 [NH, 1H], 3.75 [OCH_3 , s, 6H], 3.51 [OCH_2 , s, 2H], 1.29 [CH_3 , s, 6H].

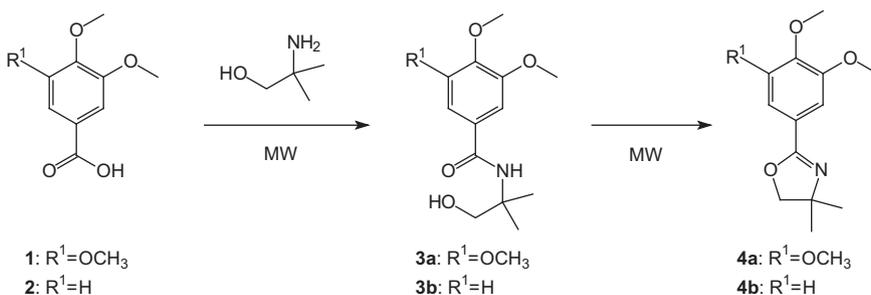
4,4-Dimethyl-2-(3,4,5-trimethoxyphenyl)-2-oxazoline (4a): yellow solid, mp 85–86°C, $\text{C}_{14}\text{H}_{19}\text{NO}_4$, MW=265.31 g mol $^{-1}$. **FTIR** (film): 3000, 2950, 2900, 2850, 1650, 1600, 1450, 1425, 1350, 1275, 1225, 1175, 1125, 1100, 850, 725. **MS** (EI, 70 eV) m/z (%): M^+ . 265 (41), 250 (100), 222 (15), 194 (21), 178 (11), 164 (3), 150 (7), 135 (7), 125 (12), 107 (4), 77 (6), 66 (7), 53 (6). $^1\text{H NMR}$ (200 MHz, CDCl_3), δ (ppm): 7.19 [(Ar-H (H-2,6), s, 2H), 4.10 [OCH_2 , s, 2H], 3.91 [OCH_3 , s, 6H], 3.88 [OCH_3 , s, 3H], 1.39 [CH_3 , s, 6H]. $^{13}\text{C NMR}$ (50 MHz, CDCl_3), δ (ppm): 161.75 [C], 152.98 [C (C-3,5)], 140.65 [C

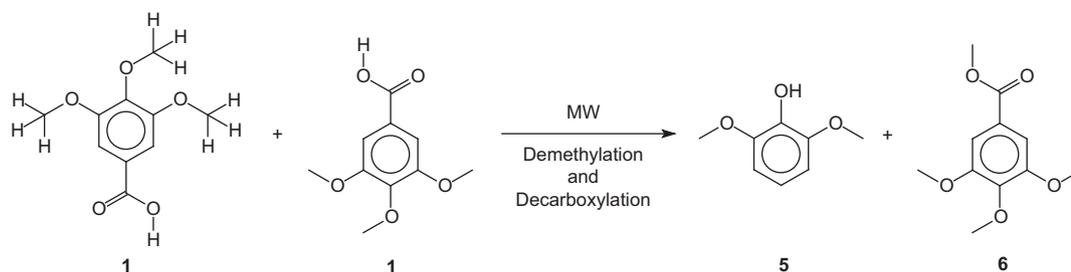
(C-4)], 123.36 [C (C-1)], 105.42 [CH (C-2,6)], 79.14 [CH_2], 67.69 [C (C-8)], 60.89 [CH_3], 56.26 [CH_3], 28.38 [CH_3].

4,4-Dimethyl-2-(3,4-dimethoxyphenyl)-2-oxazoline (4b): yellow oil, $\text{C}_{13}\text{H}_{17}\text{NO}_3$, MW=235.28 g mol $^{-1}$. **FTIR** (film): 2980, 2950, 2850, 1720, 1650, 1600, 1580, 1520, 1460, 1425, 1360, 1310, 1270, 1240, 1175, 1140, 1080, 1025, 975, 880, 820, 770, 740, 720. **MS** (EI, 70 eV) m/z (%): M^+ . 235 (34), 220 (100), 205 (5), 192 (23), 164 (26), 148 (8), 120 (5), 92 (7), 77 (8), 65 (5), 51 (3). $^1\text{H NMR}$ (200 MHz, CDCl_3), δ (ppm): 7.53 [(Ar-H (H-6), dd ($J_{6,5}=8.4$ Hz, $J_{6,2}=1.9$ Hz), 1H], 7.47 [Ar-H (H-2), d ($J_{2,6}=1.9$ Hz), 1H], 6.87 [Ar-H (H-5), d ($J_{5,6}=8.4$ Hz), 1H], 4.09 [OCH_2 , s, 2H], 3.93 [OCH_3 , s, 3H], 3.92 [OCH_3 , s, 3H], 1.38 [CH_3 , s, 6H]. $^{13}\text{C NMR}$ (50 MHz, CDCl_3), δ (ppm): 161.93 [C], 151.54 [C], 148.63 [C], 121.66 [C (C-1)], 120.68 [CH (C-5)], 110.84 [CH (C-2)], 110.39 [CH (C-6)], 79.08 [CH_2], 67.50 [C], 56.06 [CH_3], 55.94 [CH_3], 28.43 [CH_3].

2,6-Dimethoxyphenol (5): brown solid, mp 51–55°C, $\text{C}_8\text{H}_{10}\text{O}_3$, MW=154.16 g mol $^{-1}$. **MS** (EI, 70 eV) m/z (%): M^+ . 154 (100), 139 (55), 111 (20), 96 (25), 93 (35), 79 (11), 68 (12), 65 (28), 55 (11), 53 (12), 51 (15).

Methyl 3,4,5-trimethoxybenzoate (6): **MS** (EI, 70 eV) m/z (%): M^+ . 240 (60), 225 (25), 212 (39), 197 (22), 195 (21), 141 (22).

**Scheme 3** Synthesis of aryl methoxylated benzamides and 2-oxazolines.



Scheme 4 Decomposition by-products formed by microwave irradiation.

3 Results and discussion

The alternative methodology presented in this paper involves the direct reaction of carboxylic acids with amino alcohols for the synthesis of benzamides and 2-aryl-2-oxazolines. The harsh reaction conditions of classical organic synthesis were replaced by milder reaction conditions of “green” organic synthesis. The use of MW irradiation as an alternative energy source allowed the formation of the products in few min, and with excellent conversion yields. All reactions were conducted without thionyl chloride, a toxic reagent, volatile organic solvent or catalyst (Scheme 3).

As a result, the reaction allowed the isolation of a hydroxyamide intermediate, and the short reaction time contributed to the cyclodehydration process and promoted the formation of a 2-oxazoline derivative. The introduction of a methoxyl group in the aromatic ring, decreased the degree of conversion of benzamide to oxazoline [23]. The conversion rates were determined by GC-MS analyses (Table 1).

As expected, a mixture of products was also formed, including those resulting from decomposition reactions, such as demethylation and/or decarboxylation of the steric crowded carboxylic acids **1** and **2** [24], since MW irradiation easily induces decomposition reactions in function of high energy associated (Scheme 4).

In comparison with classical methods, it was verified that MW irradiation promoted the formation of benzamides and 2-oxazolines from low cost starting materials. The direct reactions were carried out in a few min, without toxic reagents, volatile organic solvents or a catalyst.

Thus, the synthesis of benzamides and 2-aryl-2-oxazolines, replacing petrochemical derivatives by compounds obtained from renewable biomass, is reported. The use of MW irradiation as an alternative energy source, allowed the establishment of milder reaction conditions, in accordance with the principles of green chemistry.

In comparison with the method recently described by Grayson [19], the synthetic procedure presents advantages such as short reaction time, despite the use of less reactive carboxylic acids, and a potential application to different amino alcohols.

The study of the reaction conditions with different carboxylic acids and amino alcohols was made, in order to establish an adequate methodology for the green synthesis of other benzamides and 2-aryl-2-oxazolines, using MW irradiation.

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