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THE EFFECT OF PYRIDINE AND TRIETHYLAMINE (TEA) CATALYSTS IN THE SYNTHESIS OF THE 4-BENZOYLOXY-3-METHOXYCINNAMIC ACID THROUGH MICROWAVE IRRADIATION METHOD

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Abstract:In this study, 4-hydroxy-3-methoxycinnamic acid and benzoyl chloride was reacted through the nucleophilic acyl substitution mechanism by using pyridine and triethylamine (TEA) as catalysts.This study aims to discern whether the 4-benzoyloxy-3-methoxycinnamic acid compound can be synthesized from 4-hydroxy-3-methoxycinnamic acid and benzoyl chloride by using pyridine and triethylamine (TEA) catalysts through microwave irradiation method. Also, this study is to discover which catalyst gives the most significant percentage of results in synthesizing 4-benzoyloxy-3-methoxycinnamic acid.This study was an experimental study, which synthesized 4-benzoyloxy-3-methoxycinnamic acid through microwave irradiation method at 180, 360, and 540 watts of power to determine the selected conditions of synthesis with pyridine catalyst.The results indicated that triethylamine (TEA) catalyst significantly gave a higher percentage of results (71.8%) than pyridine catalyst did (65.3%). The results were due to the inductive effect of ethyl on TEA and the effect of pyridine resonance associated with its basicity. The FT-IR spectrophotometry and the 1H-NMR spectrometry profile confirmed that the product of both conditions was 4-benzoyloxy-3-methoxycinnamic acid. The purity of the outcome of both reaction conditions was then determined by the melting point test and thin-layer chromatography (TLC), which revealed that the product was pure.The synthesis of 4-hydroxy-3-methoxycinnamic acid with benzoyl chloride pyridine catalyst can be carried out with microwave irradiation. Meanwhile, the synthesis with benzoyl chloride triethylamine (TEA) catalyst with microwave irradiation can only be performed at 540 watts of power. The catalyst with the more significant percentage of results, thus, is TEA catalyst with a percentage of 71.8%±1.3.

Keywords: 4-benzyloxy-3-methoxycinnamic acid, catalyst, pyridine, triethylamine, microwave irradiation.

1. INTRODUCTION

In this study, the synthesis of 4-benzoyloxy-3-methoxycinnamic acid was completed through the reaction mechanism, which was the principle of the nucleophilic acyl substitution by adding nucleophiles to C carbonyl. A nucleophile (phenolic -OH group from ferulic acid -FA) will attack C carbonyl from acyl halides (benzoyl chloride) to form tetrahedral intermediates, which will eliminate the -Cl group as leaving groups and merge with the loss of H from nucleophiles to form hydrochloric acid (HCl). In this reaction, the addition of base catalyst as pyridine to remove HCl formed during the reaction is often done (1). The catalyst will then react with HCl to form a water-soluble salt (2).

The synthesis of the 4-acetyoxy-3-methoxycinnamic acid compound with the principle of esterification reaction from acyl halide and using pyridine base catalyst through the microwave method carried out at 40 watts of powers for 0.5 minutes resulted in a percentage of 95.05%±0.71. Based on the data, it can be assumed that esterification in the phenolic -OH group of ferulic acid has been successfully carried out by using a microwave irradiation method where the percentage resulted is higher and the reaction time is more efficient (3). Catalyst is a compound that can accelerate the chemical reaction rate by decreasing the activation energy and without going through permanent changes so that a full result can be generated at the end of the reaction. Catalyst further has a significant role in synthesizing chemical compounds to obtain maximum results. On the nucleophilic acyl substitution reaction, moreover, the catalysts used are in the form of organic bases, such as pyridine and triethylamine (TEA) (1).

The synthesis of chemical compound with microwave irradiation method has been carried out plenty of times, given it can accelerate the reaction rate that the reaction can be completed faster with a more exceptional percentage results obtained and a less solvent used (4). Therefore, by referring to the results, in this study, the synthesis of 4-benzoyloxy-3-methoxycinnamic acid through the nucleophilic acyl substitution reaction with two types of catalyst (pyridine and TEA) with the microwave irradiation method is carried out. Moreover, this study aims to discern whether the 4-benzoyloxy-3-methoxycinnamic acid compound can be synthesized from 4-hydroxy-3-methoxycinnamic acid and benzoyl chloride by using both pyridine and triethylamine (TEA) catalysts with the microwave irradiation method. Besides, the other objective of this study is to ascertain, which catalyst gives a more significant percentage of the results in synthesizing 4-benzoyloxy-3-methoxycinnamic acid (5).

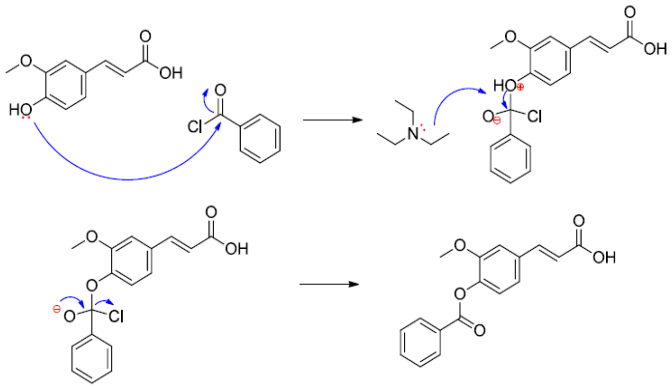

Fig. 1 The mechanism reaction on synthesis of 4-benzoyloxy-3-methoxycinnamic acid using triethylamine as catalyst

2. MATERIALS AND METHODS

This study is an experimental study that aims to discern the effect of pyridine and triethylamine (TEA) catalysts on the synthesis of 4-benzoyloxy-3-methoxycinnamic acid, ferulic acid, tetrahydrofuran (THF), benzoyl chloride, triethylamine (TEA), pyridine, and

microwave with 180, 360, and 540 watts of power in a designated time to achieve the completion of the reaction. In experimental research, replication as a validation process is indispensable, for instance, the number of experimental units that receive the same treatment under certain conditions or the same amount of treatments carried out to the experimental group with certain conditions (6).

In this study, furthermore, the replication was carried out for three times in the form of purity test, structure analysis, and the comparison of the area of both bases catalysts to know the most significant percentage of the obtained results. This study employed the Independent Samples t-Test to discover whether the synthesis of 4-benzoyloxy-3-methoxycinnamic acid by using TEA catalyst achieves a more substantial percentage of results than the one by using pyridine catalyst. The statistical test, moreover, can also be determined by observing the significance level, where Sig <0.05 indicates that Ha is accepted and Ho is rejected. On the contrary, the level where sig>0.05 suggests that Ho is accepted and Ha is rejected.

3. RESULTS

Optimization of Synthesis Reaction of 4-benzoyloxy-3-methoxycinnamic acid with Pyridine Catalyst

Reaction Completion Test

The reaction completion test was carried out by using thin-layer chromatography (TLC) with chloroform-ethyl acetate (7-1). From the optimization data, the selected reaction time was from the third sampling (6x30 seconds) at 540 watts of power. The selection was based on the visualization of relative intensity produced at the time and the power, which showed that the starting material (FA) had begun to react. This selected time was then used to determine the final percentage of the result of 4-benzoyloxy-3-methoxycinnamic acid synthesis with TLC-densitometry.

Table 1. Reaction Completion Test Result of 4-benzoyloxy-3-methoxycinnamic acid Synthesis with Pyridine Catalyst

Power (Watt)	Sampling Number	Comparison of Rf Value			Number of Impurities	Rf Value	Relative Intensity Visualization
		FA	**BA**	**BzCl**			
360	1	0.11	0.33	0.58	4	Rf1: 0.11	+++
						Rf2: 0.33	++
						Rf3: 0.55	++
						Rf4: 0.71	+++
	2	0.11	0.33	0.58	3	Rf1: 0.11	+++
						Rf2: 0.33	++
						Rf3: 0.71	+++
	3	0.11	0.33	0.58	3	Rf1: 0.11	+++
						Rf2:	++

						0.33	
						Rf3:	+++
						0.71	
	4	0.11	0.33	0.58	3	Rf1:	+
						0.11	++
						Rf2:	
						0.33	
						Rf3:	++
						0.71	
						Rf1:	++
	1	0.11	0.33	0.58	4	0.11	
						Rf2:	++
						0.33	++
						Rf3:	
						0.55	
						Rf4:	+++
						0.71	
540	2	0.11	0.33	0.58	3	Rf1:	++
						0.11	++
						Rf2:	
						0.33	
						Rf3:	+++
						0.71	
	3	0.11	0.33	0.58	3	Rf1:	+
						0.11	++
						Rf2:	
						0.33	
						Rf3:	+++
						0.71	

Notes:

- **1 : 0 x 30 seconds- 5 : 12 x 30 seconds**
- **2 : 4 x 30 seconds- FA : Ferulic Acid**
- **3 : 6 x 30 seconds- BA : Benzoic Acid**
- **4 : 10 x 30 seconds- BzCl : Benzoyl Chloride**

- **Eluent used CHCl3 : ethyl acetate = 7 : 1**
- **The appearance of impurities with ultraviolet (λ = 254 nm)**

Percentage of Synthesis Results

The data on the percentage of synthesis results of 4-benzoyloxy-3-methoxycinnamic acid with pyridine catalyst at the time of the reaction completion test above, a power of 540 Watt was selected since it has the most significant percentage of results. The chosen power was then used for the synthesis of 4-benzoyloxy-3-methoxycinnamic acid with triethylamine (TEA) catalyst (7).

Table 2. Percentage of the Results of 4-benzoyloxy-3-methoxycinnamic acid Synthesis with Pyridine Catalyst

Power (W)	Replication	Time (seconds)	Resulting Percentage (%)	Average percentage (%)

	1		**36.5%**	
180	**2**	6 x 30	**31.9%**	**31.8%±4.7**
	3		**27.1%**	
	1		**40.2%**	
360	**2**	6 x 30	**46.1%**	**45.0%±4.4**
	3		**48.7%**	
	1		**66.1%**	
540	**2**	6 x 30	**62.1%**	**65.3%±2.9**
	3		**67.7%**	

Results of 4-benzoyloxy-3-methoxycinnamic acid Synthesis with TEA Catalyst at the Selected Power (540 Watt)

The percentage of the results of 4-benzoyloxy-3-methoxycinnamic acid synthesis using triethylamine (TEA) catalyst at the selected power is shown in Table 3 below.

Table 3. Percentage of the Results of 4-benzoyloxy-3-methoxycinnamic acid Synthesis with TEA Catalyst

Power (W)	Replication	Time (seconds)	Number of Impurities	Rf Value	Resulting Percentage (%)	Average (%)
	1		**2**	Rf_1: 0.11 Rf_2: 0.71	**72.9%**	
540		6 x 30				**71.8%± 1.3**
	2		**2**	Rf_1: 0.11 Rf_2: 0.71	**70.3%**	
	3		**2**	Rf_1: 0.11 Rf_2: 0.71	**72.1%**	

Organoleptic Testing

This test was carried out to compare the organoleptic properties of the starting material (ferulic acid) to the synthesized compound. The results of the testing were obtained from the 4-benzoyloxy-3-methoxycinnamic acid compound and can be seen in Table 4 as follows.

Table 4. Results of Organoleptic Testing of 4-benzoyloxy-3-methoxycinnamic Acid Compound Results

Characteristic	Starting Materials (FA)	Resulting Compound
Shape	Solids	(Kristal) Solid (crystal)
Color	Yellowish white	White
Odor	Odorless	Quite smelly

Fig. 2 Crystal solid compound result of 4-benzoyloxy-3-cinnamic acid

Purity Test in the Determination of Melting point

The determination of melting point was carried out using the Melting Point Apparatus Electrothermal. The data indicated that the compound resulted from the synthesis had a melting point of 145-146^{o}C. In general, a compound is declared pure if the melting range is sharp with a variety of less than 2^{o}C. Therefore, it can be implied that 4-benzoyloxy-3-methoxycinnamic acid compound with pyridine catalyst is pure, based on the testing.

Table 5. Results of Melting Range Determination of 4-benzoyloxy-3-methoxycinnamic Acid with Pyridine Catalyst

Power (Watt)	Melting Range (^{o}C)			Average (^{o}C)
	Replication 1	**Replication 2**	**Replication 3**	
180	145-147	145-146	145-146	145-146
360	144-145	144-145	144-146	144-145
540	145-146	145-146	145-146	145-146
Ferulic Acid	170-172	171-173	171-172	171-172

Thin-Layer Chromatography

The Rf value of 4-benzoyloxy-3-methoxycinnamic acid synthesis with three eluent system revealed an average of 0.71, 0.63, and 0.76, respectively. Moreover, only one impurity was discovered; thus, it can be implied that the synthesized compound is pure.

Table 6. Rf Value of 4-benzoyloxy-3-methoxycinnamic Acid with Pyridine Catalyst in Purity Test using TLC

Eluent	Rf Value of Synthesized Compound			Note
	Replication I	**Replication II**	**Replication III**	
Chloroform : Ethyl acetate (7 : 1)	0.71	0.71	0.71	One impurity
Chloroform : Methanol (100 : 1)	0.64	0.64	0.62	One impurity
Dichloromethane : Ethyl acetate (5 : 1)	0.76	0.76	0.76	One impurity

Purity Test of 4-benzoyloxy-3-methoxycinnamic Acid Synthesis Results with Triethylamine (TEA) Catalyst

Determination of Melting Point

From the data, it can be noticed that commonly, the compound of synthesis results has melting ranges from 146-148ºC. In general, a compound is declared pure if the melting range is no more than 2 ºC. For that reason, it can be argued that, in this study, a 4-benzoyloxy-3-methoxycinnamic acid compound that has been synthesized with TEA catalyst is pure.

Table 7. Results of Melting Range Determination of 4-benzoyloxy-3-methoxycinnamic Acid with TEA Catalyst

Selected Power (Watt)	Melting Range (ºC)			Average (ºC)
	Replication 1	**Replication 2**	**Replication 3**	
540	146-148	146-147	147-149	146-148
Pyridine + TEA	147-148	146-148	146-148	146-148

Thin-Layer Chromatography

The Rf value of 4-benzoyloxy-3-methoxycinnamic acid compound synthesis using TEA catalyst with three eluent systems denoted the averages of 0.70, 0.63, and 0.78, respectively, with one impurity. Thus, from the results, it can be stated that the compound of the synthesis result is pure.

Table 8. Rf Value of 4-benzoyloxy-3-methoxycinnamic Acid with TEA Catalyst in Purity Test using TLC

Eluent	Rf Value of Synthesized Compound			Note
	Replication I	**Replication II**	**Replication III**	
Chloroform : Ethyl acetate (7 : 1)	0.69	0.71	0.71	One impurity
Chloroform : Methanol (100 : 1)	0.62	0.62	0.64	One impurity
Dichloromethane : Ethyl acetate (5 : 1)	0.78	0.78	0.78	One impurity

Identification of the Structure of 4-benzoyloxy-3-methoxycinnamic Acid Synthesis Results

Identification of the Structure with UV-Vis Spectrophotometer

The structure identification by implementing UV-Vis Spectrophotometer was carried out to determine the spectral pattern and the maximum wavelength of the synthesis desired compound, which was measured at a wavelength of 200-400 nm. The UV spectrum of ferulic acid, moreover, can be seen in Figure 3. In contrast, the UV spectrum of 4-benzoyloxy-3-methoxycinnamic acid with pyridine catalyst and the acid with TEA catalyst can be seen in Figure 4 and Figure 5, respectively (8).

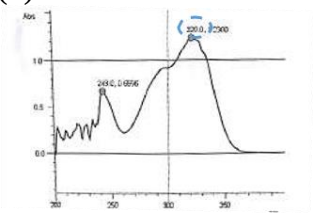

Fig. 3 The UV spectrum of ferulic acid

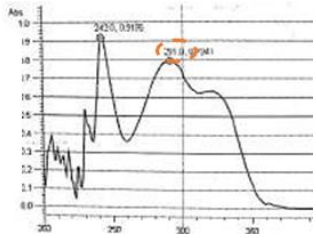

Fig.4 the UV spectrum of 4-benzoyloxy-3-methoxycinnamic acid with pyridine catalyst

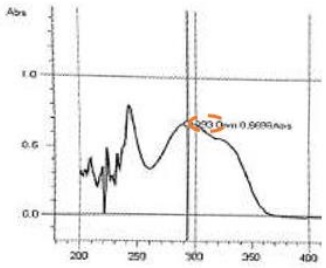

Fig.5 the UV spectrum of 4-benzoyloxy-3-methoxycinnamic acid with TEA catalyst

From the figures presented above, it can be argued that the maximum wavelength (λmax) of the resulting compounds with pyridine catalyst was 291 nm, while the one with TEA catalyst was 293 nm. These results, however, are different from the maximum wavelength of ferulic acid as the starting material, which is 320 nm.

Identification of the Structure with FR-IR Spectrophotometry

The IR spectrum of 4-benzoyloxy-3-methoxycinnamic acid synthesis with both pyridine and TEA catalysts is portrayed in Table 9 below.

Table 9. Wavenumber of each type of bond in the resulting IR spectrum from 4-benzoyloxy-3-methoxycinnamic acid synthesis

Group	Wavenumber (cm^{-1})			
	Reference	**Ferulic acid**	**Synthesis result with pyridine**	**Synthesis result with TEA**
OH (Phenolic)	3400-3200	3436	-	-
Carboxylic				
OH	3400-2400	3436	3433	3449
C=O	1730-1700	1691	1689	1735
C-O	1320-1210	1325	1288	1304
Alkene				
C-H	3100-3000	3016	3072	3079
C=C	1680-1600	1620	1629	1626
Aromatic				
C=C	1600 dan 1475	1516 dan 1432	1510 dan 1450	1513 dan 1464

C-H Esther	900-690	915	883	871
C=O Ether	1740-1715	-	1726	1780
-OCH3	2850-2810	2841	2847	2846
C-O	1300-1000	1276	1263	1253

Identification of the Structure with ^{1}H-NMR Spectroscopy

The ^{1}H-NMR spectrum of 4-benzoyloxy-3-methoxycinnamic acid is presented in the form of characteristic range interpretation, as shown in Table 10 below.

Table 10. Interpretation of H-NMR spectrum of 4-benzoyloxy-3-methoxycinnamic acid resulting compound

Chemical shift (ppm)		Multiplicity	J (Hz)	Integration	Relative position of 1H absorption	Note on H position
Theory	**Synthesis result**					
3.2-3.8	3.87	Singlet (s)	-	3H	R-OCH3	22
4.5-	6.41	Doublet (d)	16	1H	H	17
6.5	7.86	Doublet (d)	16	1H	Alkene	16
	8.189 – 8.220	Multiplet (m)	-	3H		1,2,3
	8.03	Doublet of doublets (dd)	1, 2, and 8	2H	H Aromatic	4,6
6.5-8.0						
	7.41	Triplet (t)	6	1H		12
	7.507 – 7.698	Multiplet (m)	-	2H		14,15
11.0-12.00	9.97	Singlet (s)	-	1H	R-COOH	19

Statistical Analysis Results

Based on the results of the independent-samples t-test, the significance values or Sig.(2-tailed) < 0.05 obtained for pyridine catalyst was as much as 0.024 and 0.043 for TEA catalyst. Moreover, the mean difference specified the value of -6.46667, which indicated that catalyst 1 (pyridine) has a lower average value than catalyst 2 (TEA). In other words, the percentage of the synthesis results with TEA catalyst was higher than the percentage of the results with pyridine catalyst.

4. DISCUSSION

The percentage of the results of each power (180 Watts, 360 Watts, and 540 Watts) at the selected period revealed the averages of 31.8%±4.7, 45.0%±4.4, and 65.3%±2.9, respectively. From those results, the 540 Watts of power with the highest percentage was further selected to be used in 4-benzoyloxy-3-methoxycinnamic acid synthesis with TEA catalyst. The synthesis, moreover, was carried out for 6x30 seconds and obtained an average percentage result of 71.8%±1.3. Therefore, according to the results, it can be argued that the stronger the basicity of a compound, the more the percentage of results obtained.

The determination of the purity, additionally, can be assessed from the melting range and thin-layer chromatography (TLC). The melting range of the synthesis results of 4-benzoyloxy-3-methoxycinnamic acid, furthermore, obtained an average of 145ºC-146ºC with pyridine catalyst and of 146ºC-148ºC with TEA catalyst. The melting range, as noticed, had a range of no more than 2ºC, implying that the resulting compound is pure. The purity test by implementing TLC, on the other hand, was completed by using three different eluents, namely chloroform : ethyl acetate (7:1), chloroform : methanol (100:1), and dichloromethane : ethyl acetate (5:1), which resulted in the presence of one impurity.

The structure identifications were puzzled out by employing three means, for instance, UV-Vis Spectrophotometer, FT-IR Spectrophotometry, and ^{1}H-NMR Spectroscopy. The assessment of the resulting compounds by using UV-Vis Spectrophotometer disclosed the shift in absorption towards shorter wavelengths (hypochromic). Moreover, the spectrum of the target compounds and starting materials showed different absorption patterns. The identification by using FT-IR Spectrophotometry to 4-benzoyloxy-3-methoxycinnamic acid, furthermore, obtained differences in C=O ester functional group that were not found in ferulic acid. From the result, thus, it can be assumed that the resulting compound is different from starting compound. The following identification was carried out by implementing ^{1}H-NMR Spectroscopy with a CDCl3 (Deuterated Chloroform) solvent. From the results, it was found that there were 14 protons, including protons from the -OCH3 group that had a chemical shift (δ) of 3.87 ppm in the form of singlet (3H); protons from alkenes with δ of 6.41 ppm in the form of doublets (J = 16 Hz, 1H) and 7.86 ppm in the form of doublets (J = 15.6 Hz, 1 H); protons from aromatic with δ of 7.41 ppm in the form of triplets (J = 6 Hz, 1H), δ of 7.507-7.698 ppm in the form of multiples (2H), δ of 8.03 ppm in the form of doublets (J = 1.2 and 8 Hz, 2H); δ of 8.189-8.220 ppm in the form of multiples (3H); and protons from the R-COOH group with δ of 9.97 ppm in the form of singlet (1H). If assessed from the ^{1}H-NMR spectrum between the product and the starting compound (FA), differences in terms of the number of protons from the aromatic group can be noticed. In details, five protons were found in the resulting compound at the chemical shift of 7.41 ppm to 8.22 ppm, while the starting compound only had three protons from the aromatic group at the chemical shift of 6.81 ppm to 7.14 ppm. Furthermore, on the ^{1}H-NMR spectrum of FA, it was found the protons from -OH phenolic at the chemical shift of 4.86 ppm, which was absent on the resulting compound. Thus, from those results, it can be argued that the resulting synthesis compound is indeed different from the starting compound.

5. CONCLUSION

The synthesis of 4-hydroxy-3-methoxycinnamic acid with benzoyl chloride by using pyridine catalyst can be carried out with microwave irradiation method. On the other hand, benzoyl chloride with triethylamine (TEA) catalyst can only be performed at the selected power of 540 Watts with the same process. The catalyst that obtains the most significant percentage of results in the synthesis of 4-benzoyloxy-3-methoxycinnamic acid with microwave irradiation method, thus, is TEA catalyst, with an average percentage of 71.8%±1.3.

6. ACKNOWLEDGEMENTS

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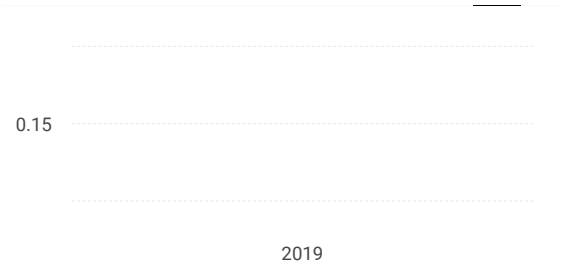

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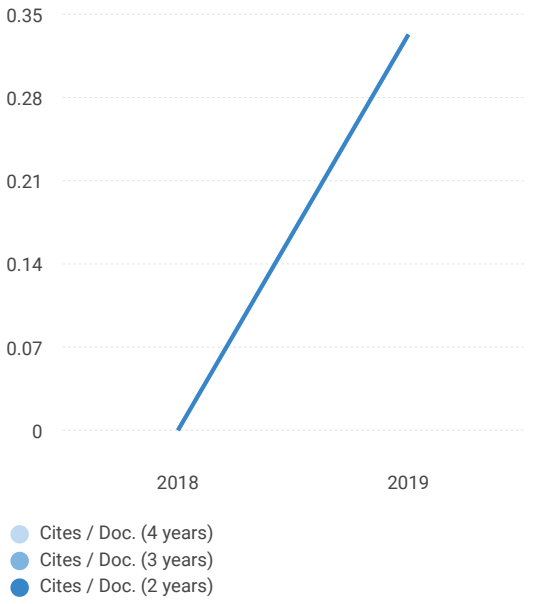

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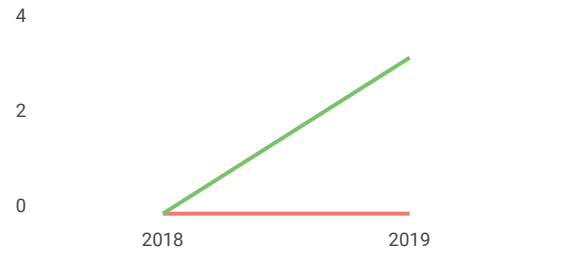

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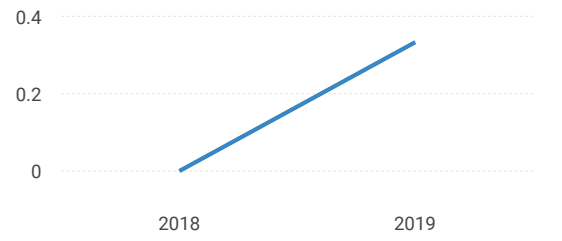

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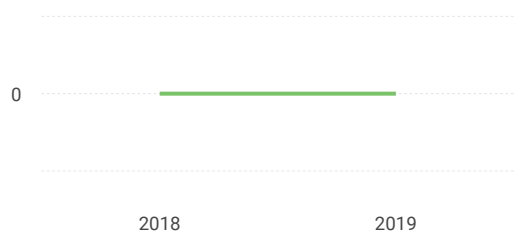

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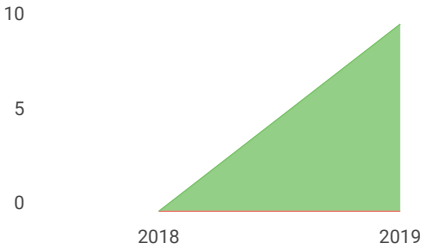

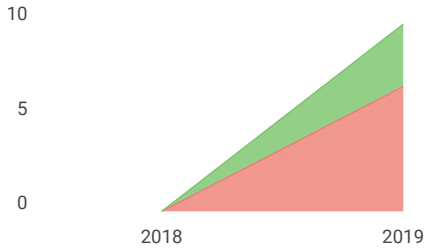

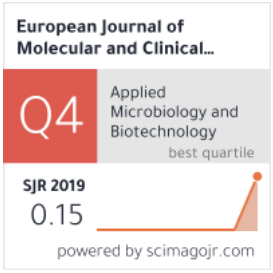

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