



## LEUCINE : AN EFFICIENT AND GREEN AMINO ACID CATALYST FOR CONVERSION OF ALDEHYDES AND KETONES INTO GEM-DIHYDROPEROXIDES WITH H<sub>2</sub>O<sub>2</sub>

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### ABSTRACT

Leucine amino acid, has been explored as an effective catalyst for conversion of ketones and aldehydes into corresponding *gem*-dihydroperoxides using 30% aqueous hydrogen peroxide in acetonitrile at room temperature. The reactions proceed smoothly within short periods of time to provide the respective *gem*-dihydroperoxides in excellent yields. Mild reaction conditions, low reaction times, high yields, low environmental impact, use of non-expensive, recyclable and green catalyst are the main merits of the present method.

### Keywords

*Gem*-dihydroperoxide; leucine; amino acid; hydrogen peroxide; aldehyde; ketone

### Academic Discipline And Sub-Disciplines

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## 1. INTRODUCTION

*Gem*-dihydroperoxides (DHPs) are considered as stable derivatives of ketones and aldehydes [1], which have been of considerable interest because of their relevance to peroxidic antimalarial drugs [2]. Also, these compounds are important intermediates in the synthesis of a number of classes of peroxides including tetraoxanes [3], silatetraoxanes [4], spirobisperoxyketals [5], bisperoxyketals [6], and 1,2,4,5-tetraoxacycloalkanes [7]. *Gem*-dihydroperoxides have also been employed as initiators for radical polymerization reactions [8], as precursors for synthesis of dicarboxylic acid esters [9], and as reagents for oxidation reactions such as epoxidation of  $\alpha,\beta$ -unsaturated ketones [10], enantioselective oxidation of 2-substituted 1,4-naphthoquinones [11], oxidation of sulfides [12], and as suitable oxidants in other synthetic organic reactions [27]. Three major methods reported for the synthesis of *gem*-dihydroperoxides are: (i) ozonolysis of ketone eneol ethers or  $\alpha$ -olefines in the presence of aqueous  $H_2O_2$  [7a, 13], (ii) reaction of ketals with  $H_2O_2$  in the presence of tungstic acid [14], or  $BF_3 \cdot Et_2O$  [15] and (iv) peroxidation of ketones using an acidic solvent [16]. However, many of these methods have certain drawbacks including the use of concentrated  $H_2O_2$  and excess acid, low yield, limited substrate range and production of mixtures of peroxidic products [17]. Also, poor selectivity and the presence of ozone sensitive groups in the substrates are further limitations in ozonolysis reaction. In order to avoid such limitations, recently, reactions of ketones and aldehydes with  $H_2O_2$  in the presence of Lewis acids in organic solvents have been reported. Amongst the Lewis acids,  $I_2$  [18], ceric ammonium nitrate (CAN) [19], CSA [20],  $NaHSO_4 \cdot SiO_2$  [21],  $Re_2O_7$  [22] and PMA [23] have been reported as the catalysts in the synthesis of *gem*-dihydroperoxides with aqueous  $H_2O_2$ . With respect to the increasing concern on the environmental issue and also to comply with the principles of the green chemistry [24], we are encouraged in the present research to examine the catalytic efficiency of leucine amino acid as a green and inexpensive catalyst for the synthesis of *gem*-dihydroperoxides from aldehydes and ketones using hydrogen peroxide. It is important to note that, so far no report on the use of amino acids as catalysts in the synthesis of *gem*-dihydroperoxides from aldehydes and ketones has appeared in the literature.

## 2. EXPERIMENTAL

### 2.1 Material and instruments

Solvents and chemicals were obtained from Aldrich and Merck chemical companies and used without purification. Melting points were determined in open capillary tubes in a Stuart SMP<sub>3</sub> apparatus and uncorrected.  $^1H$  and  $^{13}C$  NMR spectra were recorded on a JEOL FX 90Q spectrometer at 90 and 22.5 MHz respectively using  $Me_4Si$  as an internal standard. IR spectra were recorded on a Perkin Elmer GX FT IR spectrometer (KBr pellets).

*Caution:* Although we did not encounter any problem with these reactions, peroxidic compounds are potentially explosive and should be handled with precautions; all reactions should be carried out behind a safety shield inside a fume hood and transition metal salts or heating should be avoided.

### 2.2 General procedure for conversion of ketones and aldehydes into corresponding *gem*-dihydroperoxides

To a mixture of carbonyl compound **1** (1 mmol), and leucine (14 mg, 0.1 mmol) in MeCN (4 mL) was added 30% aqueous  $H_2O_2$  (2 mL), and the mixture was stirred at room temperature for an appropriate time (Table 2). After completion of the reaction as monitored by TLC, the product was extracted with chloroform (3x4 mL). Then, the combined organic layer was dried over anhydrous  $MgSO_4$ , filtered and evaporated under reduced pressure. The residue was purified by silica-packed column chromatography (hexane-EtOAc) to afford pure *gem*-dihydroperoxides (Table 2). The products were characterized on the basis of their physical and spectral ( $^1H$ ,  $^{13}C$  NMR and IR) data and compared with those reported in the literature (Table 2). The characteristic data for some representative and new products are given below.

**Methyl-(naphthalen-1-yl)-1,1-dihydroperoxide (2k).** Colorless oil; IR (KBr),  $\nu$ : 3324, 3052 (O-H stretching), 2922, 2853, 1594, 1573, 1508, 1461, 1356, 1279, 1240, 1192, 1128, 941, 863, 802, 775, 591  $cm^{-1}$ ; MS (FABMS, 70 ev):  $m/z$  (%): 243 ( $M+Na$ )<sup>+</sup>;  $^1H$ -NMR ( $CDCl_3$ , 90 MHz):  $\delta$  8.83-8.75 (brs, 2H, OOH), 8.10-7.20 (m, 7H, Ar-H), 2.66 (s, 3H,  $CH_3$ );  $^{13}C$ -NMR ( $CDCl_3$ , 22.5 MHz):  $\delta$  136.0, 134.4, 131.1, 129.9, 127.8, 126.6, 125.5, 123.0, 107.0, 20.5; *Anal.* Calcd for  $C_{12}H_{12}O_4$ : C, 65.45; H, 5.45%. Found: C, 65.42; H, 5.40%.

**(4-Bromophenyl)methylene-1,1-dihydroperoxide (2t).** Colorless solid; mp 88-90 °C; IR (KBr),  $\nu$ : 3426, 3085 (O-H stretching), 2909, 1608, 1528, 1411, 1353, 1236, 1195, 1083, 973, 856, 828, 753, 706, 601  $cm^{-1}$ ; MS (FABMS, 70 ev):  $m/z$  (%): 258 ( $M+Na$ )<sup>+</sup>;  $^1H$ -NMR ( $CDCl_3$ , 90 MHz):  $\delta$  9.96 (brs, 2H, OOH), 7.90-7.00 (m, 4H, Ar-H), 6.26 (s, 1H, CH);  $^{13}C$ -NMR ( $CDCl_3$ , 22.5 MHz):  $\delta$  141.0, 132.5, 130.5, 120.0, 111.0; *Anal.* Calcd for  $C_7H_7BrO_4$ : C, 35.74; H, 2.97%. Found: C, 35.72; H, 2.94%.

**(4-Fluorophenyl)methylene-1,1-dihydroperoxide (2u).** Colorless solid; mp 110-112 °C; IR (KBr),  $\nu$ : 3464, 3082 (O-H stretching), 2905, 1625, 1601, 1564, 1453, 1353, 1303, 1071, 1025, 844, 720, 685  $cm^{-1}$ ; MS (FABMS, 70 ev):  $m/z$  (%): 197 ( $M+Na$ )<sup>+</sup>;  $^1H$ -NMR ( $CDCl_3$ , 90 MHz):  $\delta$  9.21 (brs, 2H, OOH), 8.14-7.14 (m, 4H, Ar-H), 6.14 (s, 1H, CH);  $^{13}C$ -NMR ( $CDCl_3$ , 22.5 MHz):  $\delta$  161.62, 137.0, 128.5, 118.5, 112.0; *Anal.* Calcd for  $C_7H_7FO_4$ : C, 48.27; H, 4.02%. Found: C, 48.23; H, 3.97%.

**(2-Methoxyphenyl)methylene-1,1-dihydroperoxide (2v).** Colorless oil; IR (KBr),  $\nu$ : 3226, 3085 (O-H stretching), 2853, 1647, 1603, 1493, 1465, 1372, 1245, 1177, 1017, 844, 758  $cm^{-1}$ ; MS (FABMS, 70 ev):  $m/z$  (%): 209 ( $M+Na$ )<sup>+</sup>;  $^1H$ -NMR ( $CDCl_3$ , 90 MHz):  $\delta$  9.35 (brs, 2H, OOH), 8.18-6.84 (m, 4H, Ar-H), 6.04 (s, 1H, CH), 4.05 (s, 3H,  $OCH_3$ );  $^{13}C$ -NMR ( $CDCl_3$ ,

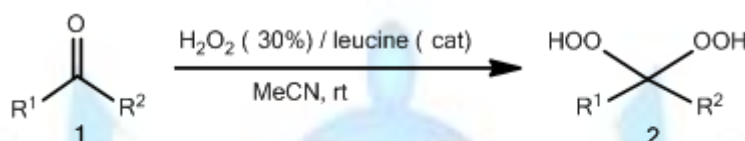
22.5 MHz):  $\delta$  157.4, 139.1, 128.5, 123.0, 118.5, 102.0, 57.5; *Anal.* Calcd for  $C_8H_{10}O_5$ : C, 51.61; H, 5.37%. Found: C, 51.57; H, 5.34%.

**(Pyridin-3-yl)methylene-1,1-dihydroperoxide (2x).** Colorless solid; Mp. > 160 °C; IR (KBr),  $\nu$ : 3410, 2941, 1635, 1450, 1393, 1139, 996, 640, 521  $cm^{-1}$ ;  $^1H$ -NMR ( $d_6$ -DMSO, 90 MHz):  $\delta$  9.75 (brs, 2H, OOH), 8.71-7.23 (m, 4H, Ar-H), 7.18 (s, 1H, CH);  $^{13}C$ -NMR ( $CDCl_3$ , 22.5 MHz):  $\delta$  150.9, 142.1, 137.6, 124.4, 118.2, 53.8; *Anal.* Calcd for  $C_6H_7NO_4$ : C, 45.86; H, 4.46; N, 8.91%. Found: C, 45.48; H, 4.42; N, 8.65%.

**(Quinolin-2-yl)methylene-1,1-dihydroperoxide (2y).** Yello solid; Mp. 138-140 °C; IR (KBr),  $\nu$ : 3425, 3250, 2958, 1660, 1629, 1600, 1375, 1355, 1111, 807, 779, 636  $cm^{-1}$ ;  $^1H$ -NMR ( $CDCl_3$ , 90 MHz):  $\delta$  8.95 (brs, 2H, OOH), 7.49-8.25 (m, 6H, Ar-H), 6.61 (s, 1H, CH); *Anal.* Calcd for  $C_{10}H_9NO_4$ : C, 57.97; H, 4.35; N, 6.76%. Found: C, 57.82; H, 4.32; N, 6.73%.

### 3. RESULTS AND DISCUSSION

In continuation of our efforts to explore new and benign catalysts for the synthesis of *gem*-dihydroperoxides [25], and their applications as versatile and high potent oxidants in various organic transformations [26], herein, we wish to introduce the leucine amino acid as a cheap, green and effective catalyst in the synthesis of *gem*-dihydroperoxides from ketones and aldehydes with 30% aqueous  $H_2O_2$  at room temperature (scheme 1).



**Scheme 1.** Leucine-catalyzed oxidative conversion of aldehydes and ketones to *gem*-dihydroperoxides with  $H_2O_2$  (30 %).

In an effort to establish the reaction conditions, various reaction parameters were studied for the preparation of 1,1-dihydroperoxycyclohexane through the model reaction of cyclohexanone with 30 % aqueous  $H_2O_2$  under the catalytic effect of leucine using different solvents such as  $CH_2Cl_2$ ,  $Et_2O$ , AcOEt,  $CH_3CN$  and the results are summarized in Table 1. As seen in this Table, the best result in terms of yield and reaction time was obtained using MeCN as the solvent of choice and 10 mol% catalyst loading at room temperature (entry 5). The importance of the catalyst in this reaction was verified by conducting the reaction in the absence of leucine that resulted in trace amount of the product (entry 10).

**Table 1.** Screening the reaction parameters for the formation of 1,1-dihydroperoxycyclohexane<sup>a</sup>

Entry	Solvent	Leucine (mol%)	Temperature (°C)	Time (min)	Yield <sup>b</sup> (%)
1	$CH_2Cl_2$	10	rt	60	70
2	$Et_2O$	10	rt	120	58
3	AcOEt	10	rt	70	87
4	EtOH	10	rt	80	85
5	$CH_3CN$	10	rt	20	96
6	$CH_3CN$	5	rt	30	75
7	$CH_3CN$	20	rt	20	92
8	$CH_3CN$	10	40	60	68
9	$CH_3CN$	10	60	60	65
10	$CH_3CN$	No catalyst	rt	120	trace

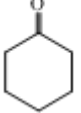
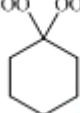
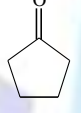

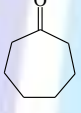
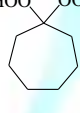

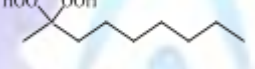
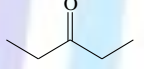
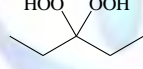
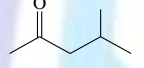
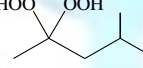
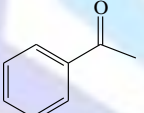
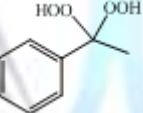
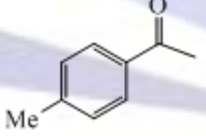
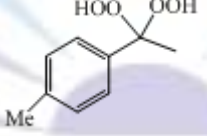
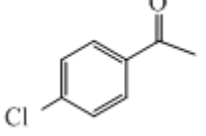
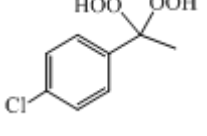
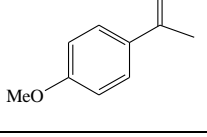
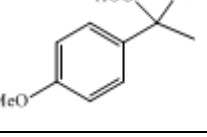
<sup>a</sup> Conditions: cyclohexanone (1 mmol), 30 % aqueous  $H_2O_2$  (3 mL), solvent (4 mL).  
<sup>b</sup> Isolated yield.

With optimized conditions in hand (aq. 30%  $H_2O_2$ , 10 mol% catalyst, MeCN, rt) we began to study the scope of the reaction using a range of aliphatic and aromatic aldehydes and ketones as summarized in Table 2. As shown in Table 2,



the aliphatic ketones **1a-f** generally react faster than the aromatic ones **1g-k** to afford the corresponding *gem*-dihydroperoxides comparatively in higher yields. Similarly, the aromatic aldehydes **1p-y** were quantitatively converted to the corresponding DHPs with relatively longer reaction times. However, this procedure proved to be unsuitable for the preparation of DHPs from the aromatic ketones **1l** and **1m** which remained untouched after *ca* 3 h reaction under the optimized conditions. This can be possibly explained by the strong resonance stabilization effects on the carbonyl group. It was interesting to note that, the addition of only one molecule of hydrogen peroxide to the carbonyl group occurs in reactions of aliphatic aldehydes such as **1n** and **1o** to result in the formation of 1,1-hydroxyhydroperoxide derivatives instead of the expected DHPs compounds.

**Table 2.** Leucine-catalyzed conversion of aldehydes and ketones into *gem*-dihydroperoxides with 30% aq. H<sub>2</sub>O<sub>2</sub><sup>a</sup>

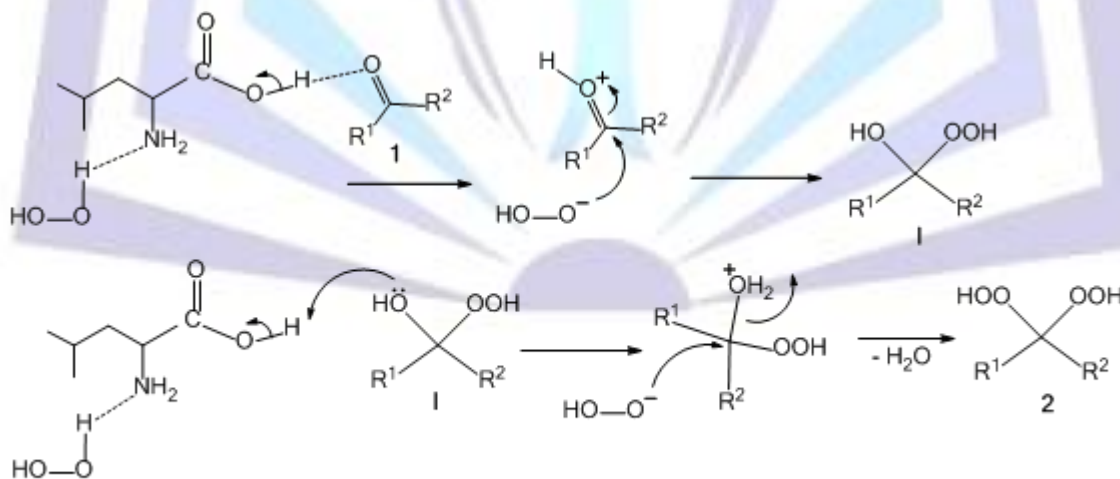
Entry	Ketone & Aldehyde <b>1</b>	Product <sup>b</sup> <b>2</b>	Time (min)	Yield (%) <sup>c</sup>	Mp (°C)	Ref.
<b>a</b>			20	96	oil	[20]
<b>b</b>			15	98	oil	[23a]
<b>c</b>			13	97	62-64	[22]
<b>d</b>			15	98	oil	[18a]
<b>e</b>			20	92	oil	[19]
<b>f</b>			18	95	oil	[19]
<b>g</b>			180	80	76-78	[19]
<b>h</b>			250	65	oil	[26h]
<b>i</b>			150	80	oil	[26h]
<b>j</b>			290	65	oil	[18b]



<b>k</b>			140	85	oil	[25d]
<b>l</b>		-	300	-	-	-
<b>m</b>		-	300	-	-	[25d]
<b>n</b>			50	95	oil	[27]
<b>o</b>			80	96	oil	[28]
<b>p</b>			30	85	oil	[19]
<b>q</b>			65	90	56-58	[18a]
<b>r</b>			100	80	oil	[23a]
<b>s</b>			50	92	74-76	[18a]
<b>t</b>			65	90	86-88	[25d]
<b>u</b>			35	86	112-114	[25d]

<b>v</b>			120	70	oil	[25d]
<b>w</b>			80	75	106-108	[25a]
<b>x<sup>new</sup></b>			20	76	> 160	-
<b>y<sup>new</sup></b>			30	88	138-140	-
<p><sup>a</sup> Conditions: ketone and aldehyde (1 mmol), CH<sub>3</sub>CN (4 mL), leucine (13 mg, 0.1 mmol), 30% aq. H<sub>2</sub>O<sub>2</sub> (3 mL), reactions are carried out at rt.</p> <p><sup>b</sup> The structures of the products were established from their physical properties and spectral (<sup>1</sup>H NMR, <sup>13</sup>C NMR and IR) analysis and compared with the data reported in the literature.</p> <p><sup>c</sup> Isolated Yield.</p>						

A reasonable mechanism to explain the conversion of ketones and aldehydes **1** into respective *gem*-DHPs **2** is given in Scheme 2. As shown in this Scheme, the initial step likely involves the activation of carbonyl compound **1** through protonation with leucine carboxyl group. Then, the protonated carbonyl undergoes nucleophilic addition with deprotonated hydrogen peroxide to produce the adduct **I**. Subsequently, the resulting hydroxyl group in the intermediate **I** is protonated by leucine carboxyl group followed by nucleophilic substitution with a second molecule of hydrogen peroxide anion to furnish the product **2**.



Scheme 2. Leucine-catalyzed synthesis of *gem*-dihydroperoxides from ketones and aldehydes.

## CONCLUSIONS

In summary, Leucine amino acid has been explored as an efficient, reusable and green catalyst which can effectively accelerate the conversion of ketones and aldehydes into their corresponding *gem*-dihydroperoxides. These reactions proceed smoothly with low reaction times at room temperature to furnish the titled products in high to excellent yields.

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