Formulation and Evaluation of Herbal Effervescent Granules Incorporated with Calliandra Haematocephala Leaves Extract

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ARTICLE INFO

Article history
Received 11/06/2013
Available online June 28/06/2013

Keywords
Calliandra haematocephala, effervescent granules, Carr’s Index.

ABSTRACT

The present research work is based on the formulation of herbal effervescent granules by incorporating the leaves extract of Calliandra haematocephala. The folklore of India widely uses this plant for treatment of various diseases and disorder. The dried leaves powder of the plant was extracted and subjected to preliminary chemical tests. Then it was formulated into effervescent granules and then evaluated for various parameters like angle of repose, dissolution studies, and effervescent cessation time. The preliminary chemical studies show that the extract contains carbohydrate, alkaloids, flavonoid, glycoside and protein. The formulated effervescent granules exhibited excellent flow properties which showed good angle of repose, Carr’s index, Hausner’s ratio, bulk density and tapped density.

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Please cite this article in press as Ramchandra Gupta et.al. Formulation and evaluation of herbal effervescent granules incorporated with calliandra haematocephala leaves extract. Indo American Journal of Pharm Research.2013:3(6).
INTRODUCTION

According to the World Health Organization traditional medicine or herbal medicine is the accumulation of the skills, knowledge and practices based on the theories, beliefs, and indigenized by different cultures, to maintain health. Nature always stands as a golden mark to exemplify the outstanding phenomena of symbiosis. Natural products from plant, animal and minerals have been the basis of the treatment of human disease [1-3]. Effervescent powders used as saline cathartics were available in the eighteenth century and were subsequently listed in the official compendia as compound effervescent powders. Effervescent mixtures have been moderately popular over the years since along with the medicinal value of the particular preparation, they offered the public a unique dosage form that was interesting to prepare. In addition, they provided a pleasant taste due to carbonation which helped to mask the objectionable taste of the drugs [4-5]. The choice of ingredients for effervescent granules depends both upon the requirement of the manufacturing process. The required ingredients are at least one acid and at least one base. The base must release carbon dioxide upon reaction with the acid. These are usually prepared from a combination of citric and tartaric acid rather than from a single acid because the use of either acid alone causes difficulties. Effervescent salts include the following ingredients, which actually produce the effervescence: sodium bicarbonate, citric acid and tartaric acid. When added to water the acids and base react to liberate carbon dioxide, resulting in effervescence [6].

The reaction between acid and Bicarbonate, which results in liberation of carbon dioxide, shown as follows:

\[
\text{A-COOH} + \text{B-HCO}_3^- \rightarrow \text{CO}_2 + \text{H}_2\text{O} + \text{B-A-COO}^- \\
(\text{Acid}) \hspace{1cm} (\text{Bicarbonate}) \hspace{1cm} (\text{Acidic Salt of Base})
\]

*Calliandra haematocephala* (Fabaceae) is a well-branched herbal plant, with height of 1 to 3 meters. Its branches are brown, cylindric, and rough and Leaves are oblong, and acute, with 5 to 10 pairs of glossy green leaflets. They had grown in gardens and parks and native to tropical America. They also found in Indian garden. Phytochemical composition of the C. haematocephala has been extensively studied in last few decades. Different parts of plant is reported to possess phenolic compounds, flavonoids, carbohydrates, alkaloids, glycosides, saponins, steroids and tannin as major phytochemical groups. This plant is traditionally used as Anti-oxidant (used as a blood purifier in some traditional medicines) [7]. EtOAC Fractionation of plant bark yielded p-hydroxybenzoic acid, protocatechuic acid, caffeic acid, astilbin, neo-isoastilbin and a non-active fraction yielded lupeol and betulinic acid. Their genus plant shows various pharmacological properties: Anti-Inflammatory, Immunomodulatory, Anticonvulsant and Antiulcerogenic. Its butanolic extract used for gastroprotective effects in acute gastric lesions [8]. They are also used traditionally for antibacterial [9].

![Figure 1: Calliandra haematocephala plant](image-url)
MATERIAL AND METHODS

Plant collection and extraction

*Calliandra haematocephala* plant was collected from the Guru Ramdas Khalsa Institute of Science and Technology (Pharmacy) college campus of Jabalpur district and it was identified by local tribes. After the collection of plant material (Leaves), it was dried in shade for 24 hrs and the leaves were reduce in small size and pass through the sieve of No. 40. The 500g of powered plant material was defatted with petroleum ether and then extracted with Methanol for 48 h at 60°C. The extract thus obtained was then concentrated under vacuum using rotary vacuum evaporator and then subjected to preliminary chemical screening to identify the active chemical constituents.

Phytochemical screening

The leaves extract of *C. haematocephala* was collected in beaker. Preliminary tests were carried out for the presence or absence of phyto-constituents like Glycosides, Flavanoids, Saponins, Alkaloids, Carbohydrates, Sterols, Proteins, Phenolic compounds and reducing compounds. A description of methods adopted for performing the tests were carried out by made procedure [10-11].

Formulation of herbal effervescent granules

Herbal effervescent granules were prepared by wet granulation method. The *Calliandra haematocephala* leaves extract (active ingredient) 7.50 mg, Talc powder 7.50 mg, magnesium esterase 3.75 mg, saccharin 73.50 mg, polyethylene glycols (PEG) 12.00 mg, citric acid 79.33 mg, tartaric acid 158.66 mg, sodium bicarbonate 269.71 mg and polyvinyl pyrrolidone (PVP) binder 24.00 mg. The extract was dried in oven at 60°C to constant weight and triturated in a mortar and pestle to make powder then mixed with calculated amount of the other components. The binder was added and formed into a paste and granulated using sieve No. 40. Then sufficient alcohol was added to make a damp mass. This mass was passed through sieve No. 20 to get granules and these granules were dried in hot air oven at 40°C and then they were packed in air tight container [2-4-5].

![Figure 2: Herbal Effervescent Granules of Calliandra haematocephala](image)

EVALUATION OF FORMULATED HERBAL EFFERVESCENT GRANULES [12-18]

1. Angle of Repose

The fixed funnel method was employed to measure the angle of repose. A funnel was secured with its tip at a given height (h), above a graph paper that is placed on a flat horizontal surface. The blend was carefully pored through the funnel until the apex of the conical pile just touches the tip of the funnel. The radius of the base of the conical pile was measured. The angle of repose (θ) Was calculated using the following formula:

\[ \tan \theta = \frac{h}{r} \]
Where, \( \theta \) = Angle of repose, \( h \) = Height of the cone, \( r \) = Radius of the cone base. Values for angle of repose \( \leq 30^\circ \) usually indicate a free flowing material and angles \( \geq 40^\circ \) suggest a poorly flowing material, 25-30 show excellent flow properties, 31-35 show good flow properties, 36-40 show fair flow properties and 41-45 showing passable flow properties.

2. **Bulk Density**

15 g powder blend introduced into a dry 100 ml cylinder, without compacting. The powder was carefully leveled without compacting and the unsettled apparent volume, \( V_0 \), was read. The bulk density was calculated using the following formula.

\[
\rho_b = \frac{M}{V_0}
\]

Where, \( \rho_b \) = Apparent bulk density, \( M \) = Weight of sample, \( V \) = Apparent volume of powder.

3. **Tapped Density**

After carrying out the procedure as given in the measurement of bulk density the cylinder containing the sample was tapped 500 times initially followed by an additional taps of 750 times until difference between succeeding measurement is less than 2\% and then tapped volume, \( V_f \) was measured, to the nearest graduated unit. The tapped density was calculated, in gm per ml, using the following formula.

\[
\rho_{tap} = \frac{M}{V_f}
\]

Where, \( \rho_{tap} \) = Tapped density, \( M \) = Weight of sample, \( V_f \) = Tapped volume of powder.

4. **Carr’s index**

The Compressibility index (Carr’s index) is a measure of the propensity of a powder to be compressed. It is determined from the bulk and tapped densities. In theory, the less compressible a material the more flowable it is. As such, it is measures of the relative importance of interparticulate interactions. In a free flowing powder, such interactions are generally less significant, and the bulk and tapped densities will be closer in value. For poorer flowing materials, there are frequently greater inter-particle interactions, and a greater difference between the bulk and tapped densities will be observed. These differences are reflected in the Carr’s Index which is calculated using the following formulas:

\[
\text{Compressibility index} = \left( \frac{\rho_{tap} - \rho_b}{\rho_{tap}} \right) \times 100
\]

Where, \( \rho_b \) = Bulk Density, \( \rho_{tap} \) = Tapped Density.

**Table 1:** Compressibility index values

<table>
<thead>
<tr>
<th>Compressibility index</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \leq 10 )</td>
<td>Excellent</td>
</tr>
<tr>
<td>11 – 15</td>
<td>Good</td>
</tr>
<tr>
<td>16 – 20</td>
<td>Fair</td>
</tr>
<tr>
<td>21 – 25</td>
<td>Passable</td>
</tr>
<tr>
<td>26 – 31</td>
<td>Poor</td>
</tr>
<tr>
<td>32 – 37</td>
<td>Very Poor</td>
</tr>
<tr>
<td>&gt;38</td>
<td>Very Very Poor</td>
</tr>
</tbody>
</table>

5. **Hausner’s Ratio**

Hausner’s ratio is an indirect index of ease of powder flow. It is calculated by the following formula.

\[
\text{Hausner’s Ratio} = \frac{\text{Tapped density (pt)}}{\text{Bulk density (pb)}}
\]

Where, \( pt \) tapped density and \( pb \) is bulk density. Lower Hausner’s ratio (<1.25) indicates better flow properties than higher ones, between 1.25 to 1.5 showing moderate flow properties and more than 1.5 poor flow.
6. **Effervescent Cessation Time**

100 ml of distill water was taken in 250ml beaker, one dose of effervescent granules were poured in to the beaker, effervescent cessation time and effervescent production was observed.

**RESULTS AND DISCUSSION**

The Methanolic extract of *C. haematocephala* after extraction gave yield of 28.5% w/w. When subjected to preliminary chemical screening showed the presence of Flavonoids, Carbohydrate, Glycoside, Protein and Alkaloids. An herbal effervescent granule was prepared as this particular formulation can be easily administered by all age groups. The colour of the granules was olive green with characteristic odor. The angle of repose of granules was 30.5. Bulk density ($\rho_b$), and tapped density ($\rho_{tap}$) were 0.52 and 0.68 respectively. The Compressibility index (Carr’s index) was 23.5 and Hausner ratio was 1.31 which shows its moderate flow property. All results are showed in table 2 and 3.

**Table 2**: Phytochemical screening of Methanolic extract of leaves of *C. haematocephala*.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Phytoconstituent</th>
<th>Test</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Carbohydrate</td>
<td>Benedicts and Fehling</td>
<td>+</td>
</tr>
<tr>
<td>2</td>
<td>Protein</td>
<td>Biuret test</td>
<td>+</td>
</tr>
<tr>
<td>3</td>
<td>Steroid</td>
<td>Salkowski test</td>
<td>+</td>
</tr>
<tr>
<td>4</td>
<td>Glycosides</td>
<td>Legal’s test</td>
<td>+</td>
</tr>
<tr>
<td>5</td>
<td>Flavanoids</td>
<td>Shinoda test</td>
<td>+</td>
</tr>
<tr>
<td>6</td>
<td>Saponin</td>
<td>Foam test</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>Alkaloids</td>
<td>Dragon dorff’s and wagner’s test</td>
<td>+</td>
</tr>
<tr>
<td>8</td>
<td>Tannin</td>
<td>Lead acetate and acetic acid test</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>Fat</td>
<td>Filter paper test</td>
<td>-</td>
</tr>
</tbody>
</table>

**Table 3**: Physical Evaluation of Granules

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Parameter</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Angle of Repose</td>
<td>30.5</td>
</tr>
<tr>
<td>2</td>
<td>Bulk density</td>
<td>0.52</td>
</tr>
<tr>
<td>3</td>
<td>Tapped density</td>
<td>0.68</td>
</tr>
<tr>
<td>4</td>
<td>Carr’s index</td>
<td>23.5</td>
</tr>
<tr>
<td>5</td>
<td>Hausner ratio</td>
<td>1.31</td>
</tr>
<tr>
<td>6</td>
<td>Effervescent Cessation Time</td>
<td>2-3 min.</td>
</tr>
<tr>
<td>7</td>
<td>Color</td>
<td>Olive green color</td>
</tr>
<tr>
<td>8</td>
<td>Odor</td>
<td>Characteristic odor of <em>Calliandra haematocephala</em></td>
</tr>
<tr>
<td>9</td>
<td>Apperance</td>
<td>Amorphous Granules</td>
</tr>
</tbody>
</table>

**CONCLUSION**

The leaves extract of *Calliandra haematocephala* was found to contain Phytochemical constituent likes glycoside, alkaloids, flavonoid, protein and carbohydrate which are the active ingredients drug. Effervescent granules were formulated from the Methanolic leaves extract of *Calliandra haematocephala* and optimized using different granules additives for convenient oral administration granules. Hence it can be suggested that the formulated herbal effervescent granules can be a very good alternative as it has proved to be very effective for their activity. The formulated granules were subjected to the known official monographs requirements and were found to comply with the standards of the BP and IP. These granules which were prepared from local plant that
grows wild in India and other parts of world can be used as an effervescent drug with low price and short disintegration time.

REFERENCE