Objective: The objective of this research was to formulate Orally Disintegrating Tablets (ODT) preparations for secang (Caesalpinia sappan L.) wood extract as antioxidants that met the requirements as a pharmaceutical preparation.

Methods: Three ODT formulas were made using the composition of the extract of secang wood, HPC-H, Kollidon® CL, Avicel® PH 101, mannitol, and so on. The bitter taste of the extract of wood covered was secured with the Hydroxy Propyl Cellulose High Substitution (HPC-H) masking agent. Variations in HPC-H concentration were 4%, 4.5%, and 5%. The evaluation of tablet print mass tested included water content, flow velocity, and resting angle, real density, incompressible density, and compressibility. Evaluation of tablets tested included weight uniformity, uniformity of size, hardness, friability, and disintegration time. The tablet antioxidant activity testing was carried out by the Diphenylpicrylhydrazyl (DPPH) method.

Results: The results showed that all ODT formulas of secang wood extract were fulfilling the requirements as a tablet preparation according to Indonesian Pharmacopoeia IV. IC_{50} values obtained from antioxidant testing on the three ODT formulas for a secang wood extract for formula I was 3.614 ppm, formula II was 3.464 ppm, formula III was 3.173 ppm, and the wood extract was 3.757 ppm.

Conclusion: The results obtained in this research work clearly indicated ODT formulas of secang wood extract fulfilled Indonesian Pharmacopoeia IV requirement and belong to the category of very strong antioxidants because they have an IC_{50} value of less than 50 ppm.

Keywords: Secang wood, Caesalpinia sappan, Antioxidant, Orally Disintegrating Tablets (ODT), DPPH
sesquiterpenoids in secang wood and its extract guidance by Farnsworth method [12].

**Standardization of extracts**

Standardization of wood extract was carried out to ascertain water content, drying losses, levels of soluble ethanol extracts, levels of. Water-soluble extracts, total ash content, acid insoluble ash content and metal content of extracts guided by the Indonesian Pharmacopeia 4th edition [13].

The antioxidant activity test was determined by the UV-visible spectrophotometric method using DPPH (1, 1-diphenyl-2-picryl-hydrazyl) reagent by looking at the absorbance of the secang wood extract in the extract and in ODT. Vitamin C was used to control positive. This method was similar to other published works [16-18] and prepared with the following: making a secang wood extract solution, making DPPH solution, determination of the maximum wavelength of DPPH, determination of DPPH operating time, determination of operating time DPPH with extract, testing the antioxidant activity of extracts and ODT of secang wood extract. The blank used was the extract solution and the solution of each formula with the same concentration. The antioxidant activity of the test solution was calculated by the following formula:

\[
\% \text{ inhibition} = \left[ 1 - \frac{A_{\text{test}}}{A_{\text{control}}} \right] \times 100\%
\]

The IC50 value was calculated from the linear regression curve between the percent inhibition of absorbance to the concentration of extract and ODT of secang wood extract.

**Phytochemical screening**

Phytochemical screening results can be seen in table 2. Chandra [20] reported that C. sappan showed the presence of flavonoids, phenolic compounds, tannins, saponin, protein, oxalic acid, carbonate, oil, and fat. Preliminary phytochemical screening of C. sappan leaves showed the presence of carbohydrates, glycosides, flavonoids, phenols, tannins and amino acids in the crude drug was reported by Kaur et. al [21]. Srinivasan et. al [22] in their preliminary phytochemical analyses showed the presence of steroids, tannin, phenol, saponins, and flavonoids in the ethanolic extract. This slight difference in phytochemical screening results was most likely due to the origin of the sample.

**Results and discussion**

The hedonic test [19] was carried out on 30 panelists taken randomly. Panelists were asked to taste one ODT extract of wood extract from each formula, then asked for a response and assessment of the tablet through a given questionnaire, including the level of preference for the tablet, preference for taste, preference for after taste, and preference for the level of irritation. Data analysis: carried out using the Newman-Keuls statistical test.

**Antioxidant activity measurement**

In this study, three formulas were made using the composition of the extract of secang wood, HPC-H, kollidon® CL, Avicel® PH 101, mannitol, acesulfame, magnesium stearate, citric acid, and menthol. Tablet formulations can be seen in table 1. Formulation testing referred to other ODT tablet published by Liang and Chen [14]. Table evaluation included weight uniformity, uniformity of size, uniformity of hardness, fragility (friability), and disintegration time [15].

**ODT formulation**

Table 1: ODT secang wood extract formula

| Composition                | Formula (mg) |
|----------------------------|--------------|
|                            | F1   | F2   | F3   |
| Secang wood extract        | 100.00| 100.00| 100.00|
| HPC-H                     | 20.00 | 22.50 | 25.00 |
| Kollidon® CL              | 20.00 | 20.00 | 20.00 |
| Avicel® PH 101            | 237.25| 234.75| 232.25|
| Mannitol                  | 90.00 | 90.00 | 90.00 |
| Acesulfame                | 15.00 | 15.00 | 15.00 |
| Magnesium Stearate        | 10.00 | 10.00 | 10.00 |
| Citric acid               | 7.50  | 7.50  | 7.50  |
| Menthol                   | 0.25  | 0.25  | 0.25  |
| Total                     | 500.00| 500.00| 500.00|

Notes: + = detected; - = not detected

Table 2: Secondary metabolites of Caesalpinia sappan

| No | Secondary metabolites | Wood sample | Extract |
|----|-----------------------|-------------|---------|
| 1  | Alkaloids             | +           | +       |
| 2  | Flavonoids            | +           | +       |
| 3  | Quinine               | +           | +       |
| 4  | Monoterpenoids and Sesquiterpenoids | + | + |
| 5  | Polyphenol            | +           | +       |
| 6  | Saponins              | -           | -       |
| 7  | Steroids and Triterpenoid | -       | -       |
| 8  | Tannin                | +           | +       |

**Hedonic taste**

To find out the quality of the secang wood extract used more clearly can be seen in table 3.

**Standardization of extracts**

**Table 3: Quality tests ODT secang wood**

| No | Tests                | Results   |
|----|----------------------|-----------|
| 1  | Weight uniformity    | Uniform   |
| 2  | Uniformity of size   | Uniform   |
| 3  | Uniformity of hardness| Uniform   |
| 4  | Fragility (friability)| Uniform   |
| 5  | Disintegration time  | Uniform   |
obtained did not show the detection of the three metals in the 0.024 μg/kg and 13.72 mg/kg, and the Sn, Cd, and As metals carried out, it was obtained that the metal content of Hg and Pb was extracted sappan wood. The value of Hg metal detected in the secang National Standard in 2009, which is equal to 0.1 mg/kg. Meanwhile, in 2009 [26], which was 7.0 mg/kg. The high value of Pb metal the value of the Pb metal levels detected in the extract of secang effective controlled release (CR) material since it was a hydrophilic Grade of Hydroxypropyl Cellulose (HPC- H) was thought as an content detected in the wood extract was caused by environmental contamination during planting due to environmental factors. From the results of metal assays using Shimadzu AA7000 that had been carried out, it was obtained that the metal content of Hg and Pb was 0.024 μg/kg and 13.72 mg/kg, and the Sn, Cd, and As metals obtained did not show the detection of the three metals in the extract sappan wood. The value of Hg metal detected in the secang wood extract was still below the limit stated in the Indonesian National Standard in 2009, which is equal to 0.1 mg/kg. Meanwhile, the value of the Pb metal levels detected in the extract of secang wood was above the limit stated in the Indonesian National Standard in 2009 [26], which was 7.0 mg/kg. The high value of Pb metal content detected in the wood extract was caused by environmental influences.

Formulation

In the research that had been done, DTF secang wood extract was made in 3 formulations with different concentrations in the masking agent, namely HPC-H with a concentration variation of 4.0%, 4.5%, and 5.0 %. (see table 1). The composition considerations were designed based on the following considerations. High Viscosity Grade of Hydroxypropyl Cellulose (HPC-H) was thought as an effective controlled release (CR) material since it was a hydrophilic polymer. It was for hydrophilic matrix and sustained release formulation[27]. It dissolved in water and organic solvents, at room temperature, inert with regard to the active ingredients (API), improved the hardness of the tablet, optimized disintegration of the tablet, was soluble in aqueous mediums and 100% ethanol. Due to the balance of strong disintegration and optimal surface homogeneity, our cross-povidone Kollidon® CL-F was our first choice tablet disintegrant. The super-disintegrant was specifically suitable for use in small tablets to fasten disintegration and dissolution. Kollidon® CL-F could also be used in wet granulation processes when a large number of granulation liquids were needed [28]. Avicel® PH 101 was used as a disintegrating agent [29]. Mannitol [30] was a polysaccharide alcohol and an isomer of sorbitol. Mannitol (C6H12O16) was used in pharmaceutical products as a sweetening agent, tablet and capsule diluent, excipient for chewable tablets, a toxicity agent, and as a vehicle (bulk agent) for lyophilized preparations. Acetarsulfate potassium was often used in sweetener blends to produce a more sugar-like taste in a food or beverage. The ingredient also helped the blend retain its sweetness during baking or heat processing, which was important for preparing foods, such as cookies and candies [31]. Magnesium stearate was a fine white powder. Its main purpose was providing a lubricant for capsules and tablets. It increased flowability, which ensured efficiency through the manufacturing process [32]. Citric Acid Used in Food. About 50 percent of the world’s citric acid production was used as a flavor enhancer in beverages. Citric acid created a slightly tart, refreshing flavor and balances the sweetness in sodas, teas, juices, and other drinks. The acid pH of citric acid also made it useful as a preservative [33]. Menthol was alcohol produced from mint oils or prepared synthetically. When added to pharmaceuticals and foods, menthol functions as a fortifier for peppermint flavors. It also had a counterirritant effect on skin and mucous membranes, thereby producing a local analgesic or anesthetic effect [34].

Print process in process control (IPC)

To find out the nature of the substance to be printed, IPC was tested for print mass. This test was intended to determine whether the print mass was feasible or not so that this test could be used as a supporting factor to determine the quality of the tablet. To be more clear, it can be seen in table 4.

Table 3: Results of standardization of extracts

| Tests                        | Concentration (%) | Concentration based on indonesian herbal pharmacopeia (%) [23] |
|------------------------------|-------------------|-----------------------------------------------------------------|
| Water-soluble extract        | 19.5±0.76376      | -                                                               |
| Ethanol soluble extract      | 40.5±0.076376     | -                                                               |
| Water content                | 0.5±0.05          | <0.1                                                            |
| Drying shrinkage             | 0.5±0.05          | -                                                               |
| Total ash content            | 9.22±0.251661     | 1.4                                                             |
| Acid-insoluble ash content   | 4.56±0.3          | <0.6                                                            |

From the results of standardization of extracts that had been done, it was found all parameters fulfilled the concentration described in Indonesian Herbal Pharmacopeia. Actually, provisions regarding the need for standardization of extracts from herbal ingredients are also required by BBOT (Traditional Medicine Raw Materials) [24] and WHO [25]. The water content obtained at 0.5% meaning it could reduce the risk of damage to secang wood extract due to the growth of fungi and bacteria. In addition, the value of water content was also related to the dosage on the preparation because the extract with a high value of the water content, the amount of extract needed to reach the dosage on more preparations. The results obtained from testing drying losses were 0.5%. It indicated that the compounds in the secang wood extract were only lost or evaporated as much as 0.5% during the heating process. The results of total ash content determination were 9.22% and the results of the determination of acid insoluble ash content were 4.56%. These results were not in accordance with the standards listed in Herbal Pharmacopoeia. This was possibly due to metal or mineral contamination during planting due to environmental factors. From the results of metal assays using Shimadzu AA7000 that had been carried out, it was obtained that the metal content of Hg and Pb was 0.024 μg/kg and 13.72 mg/kg, and the Sn, Cd, and As metals obtained did not show the detection of the three metals in the extract sappan wood. The value of Hg metal detected in the secang wood extract was still below the limit stated in the Indonesian National Standard in 2009, which is equal to 0.1 mg/kg. Meanwhile, the value of the Pb metal levels detected in the extract of secang wood was above the limit stated in the Indonesian National Standard in 2009 [26], which was 7.0 mg/kg. The high value of Pb metal content detected in the wood extract was caused by environmental influences.

Table 4: Print mass evaluation results

| Parameter                  | Formula 1          | Formula 2       | Formula 3        | Requirement [35] |
|----------------------------|-------------------|----------------|-----------------|-----------------|
| Drying Loss (%)            | 1.49±0.0235       | 1.45±0.130     | 1.63±0.145      | <2              |
| Flow Rate (g/s)            | 11.81±0.339       | 11.68±0.179    | 11.58±0.554     | >10             |
| Break Angle (°)            | 21.02±0.500       | 22.96±1.132    | 20.58±1.446     | <25             |
| Real Density (g/ml)        | 0.453±0.005       | 0.442±0.005    | 0.551±0.003     | -               |
| Compressibility density (g/ml) | 0.517±0.008     | 0.519±0.007    | 0.568±0.005     | -               |
| Compressibility (%)        | 16.228±1.049      | 14.948±0.963   | 15.542±0.077    | <20             |

From the results of testing for shrinkage drying, all formulas met the requirements. The results of the drying shrinkage test could affect the powder flow rate because it related to the moisture from the tablet print mass. If the tablet’s print mass had high humidity, it would affect the tablet printing process by sticking the tablet to the punch, also that the tablet produced was more fragile. From the results of testing the flow rate that had been done, the flow properties of all formulas were good because they were more than 10 g/s [35]. This was related to the tablet printing process. The better the mass flow rate of the print, the easier the mass could flow when printing tablets. Tests for resting angles were also carried out from data obtained after flow rate testing was carried out. The results of the resting angle test obtained from all formulas were very good because the values show less than 250 [35]. The greater the value of the resting angle, the worse the print mass flow. This could cause unevenness in the weight of tablets produced. From the results of compressibility testing that had been done on all formulas, the value of compressibility was good because it was less than 20 %. Based on the results of compressibility testing that had been done it could be concluded that the process of compressing the print mass could be done because of the nature of the flow of powder and the ability of the print mass to form a preparation would be stable and compact when given pressure.
ODT quality control (QC)
After the process of evaluating the print mass, the printed mass of the tablet was made using the direct press method. The tablets produced were evaluated according to the requirements of the Indonesian Pharmacopoeia IV Table 5: ODT quality control

| Parameter           | F1               | F2               | F3               |
|---------------------|------------------|------------------|------------------|
| Weight (mg)         | 506±7.345        | 516±7.896        | 511±7.443        |
| Diameter (mm)       | 12.137±0.036     | 12.160±0.013     | 12.138±0.023     |
| Thickness (mm)      | 5.226±0.037      | 5.3125±0.077     | 5.349±0.060      |
| Hardness (N)        | 53.3±6.672       | 49.125±5.458     | 40.875±5.636     |
| Friability (%)      | 0.324±0.163      | 0.577±0/324      | 0.796±0.156      |
| Disintegration Time (seconds) | 30.667±6.506   | 26.667±5.312     | 18.333±3.055     |

Results of the antioxidant activity
Antioxidant activity test was carried out to determine the antioxidant activity in secang wood extract and ODT extract of secang wood extract. The antioxidant activity test was carried out by determining the 50% inhibitory concentration (IC50) from the secang wood extract and ODT preparation for secang wood extract using the DPPH (1,1-diphenyl-2-picrylhydrazyl) method with a UV-Visible spectrophotometer. In this study, vitamin C was used as a comparative antioxidant.

Antioxidant activity
In this test, the antioxidant activity of ODT extract of secang wood was tested. Each ODT formula contains 100 mg of secang wood extract. The test solution was made by weighing one tablet from each formula and dissolved in 100 ml of 96% ethanol so that the solution concentration of 100 ppm was obtained. After that, the solution was centrifuged to separate the extract from the tablet filling component. The supernatant obtained was then diluted to a concentration of 20 ppm. After that, multilevel dilution was carried out with variations in concentration, namely 8 ppm, 4 ppm, 2 ppm, 1 ppm, and 0.5 ppm. Then DPPH solution was added with a concentration of 40 ppm into each test solution and incubated for 40 min. After incubation, absorbance measurements were carried out with a UV-Visible spectrophotometer on each of the test solutions.

Results obtained from testing the antioxidant activity of each ODT extract of the wood extract are shown in table 6.

Table 6: Antioxidant activity results

| Test Sample     | Concentration (ppm) | % Inhibition | Linear regression equation | IC50 (ppm) |
|-----------------|---------------------|--------------|-----------------------------|------------|
| Extract         | 8                   | 71.5973±4187 | Y = 5.142X+30.68            | 3.757      |
|                 | 4                   | 51.53066809  | R = 0.999                   |            |
|                 | 2                   | 41.43980238  | R = 0.999                   |            |
|                 | 1                   | 35.86020182  | R = 0.999                   |            |
|                 | 0.5                 | 32.69341209  | R = 0.999                   |            |
|                 | 3                   | 57.42297138  | R = 0.999                   |            |
|                 | 2.5                 | 53.27966607  | R = 0.999                   |            |
| Vitamin C       | 2                   | 48.70304114  | Y = 9.096X+30.27            | 2.169      |
|                 | 1                   | 38.35718545  | R = 0.997                   |            |
|                 | 0.5                 | 35.49493143  | R = 0.997                   |            |
| Tablet Formula 1| 8                   | 62.86860582  | Y = 3.129X+38.63            | 3.614      |
|                 | 4                   | 55.01905717  | R = 0.995                   |            |
|                 | 2                   | 44.92276830  | R = 0.995                   |            |
|                 | 1                   | 40.07010632  | R = 0.995                   |            |
|                 | 0.5                 | 40.18054162  | R = 0.995                   |            |
| Tablet Formula 2| 8                   | 67.09215542  | Y = 4.021X+36.07            | 3.464      |
|                 | 4                   | 55.19925280  | R = 0.989                   |            |
|                 | 2                   | 43.29078456  | R = 0.989                   |            |
|                 | 1                   | 38.46513076  | R = 0.989                   |            |
|                 | 0.5                 | 40.36108325  | R = 0.989                   |            |
| Tablet Formula 3| 8                   | 82.44175210  | Y = 6.564X+29.17            | 3.173      |
|                 | 4                   | 52.73066170  | R = 0.985                   |            |
|                 | 2                   | 35.07921715  | R = 0.985                   |            |
|                 | 1                   | 32.32095713  | R = 0.985                   |            |
|                 | 0.5                 | 35.49493143  | R = 0.985                   |            |
From the results of the activity curves of each tested sample obtained a regression equation \( y = bx + a \), then entered the value of 50 substituted into \( y \), the IC\(_{50} \) value was obtained. IC\(_{50} \) value was the concentration value where the sample was able to reduce DPPH activity by 50% from the initial concentration. The results of IC\(_{50} \) value calculation showed IC\(_{50} \) extract value of 3.757 ppm, tablet formula 1 was 3.614 ppm, tablet formula 2 was at 3.464 ppm, tablet formula 3 at 3.173 ppm. Vitamin C used as a comparison in this test has an IC\(_{50} \) Value of 2.169 ppm. Fig. 1 shows a summary of these IC\(_{50} \) values.

In fig. 1 the IC\(_{50} \) value of secang wood extract with the IC\(_{50} \) value of the three ODT formulas of secang wood extract had different values, this was presumably due to the influence of additives in tablet formulations, such as citric acid which increased the antioxidant activity in the formula IC\(_{50} \) on the extract, because citric acid had activity as an antioxidant. Thus, the IC\(_{50} \) value in all formulas was greater than the extract. A compound was said to be an antioxidant very strong if the IC\(_{50} \) was less than 50 ppm. Strong if IC\(_{50} \) values range from 50-100 ppm, medium if IC\(_{50} \) values range from 100-150 ppm, weak if IC\(_{50} \) values range from 150-200 ppm [38]. Judging from the results obtained, all the ODT formulas of the secang wood extract could be classified in the category of very strong antioxidants because the IC\(_{50} \) value obtained was less than 50 ppm. By using DPPH and nitric oxide methods, strong antioxidant properties of C. sappan ethyl acetate, methanol, and water extracts were reported by Badami et al. [39-41]. Formulation of Liposome using Sappanwood (Caesalpinia Sappan L.) ethyl acetate fraction as an activate compound had been reported by Noviza et al. [42].

**Hedonic taste results**

The preference test was carried out on all ODT extracts of secang wood extract that met the demolition requirements for 30 panelists. Test results for ODT preferences for secang wood extract included levels of preference, taste, after taste, and degree of deformity. From the results of the test of preference made on the level of preference, there were differences in the level of preference of panelists on the ODT tablets of secang wood extract with differences in the level of preference of the panelists that were evident between the criteria of dislike, rather dislike, neutrality, likes rather like criteria [19]. From the results of the preference test conducted on after taste, it was found that there were differences in panelists' judgments about after taste, it was found that there were differences in panelists' assessment of after taste against ODT tablets secang wood extract with differing panelists' judgments regarding real after taste between very bitter criteria and criteria neutral, rigid, and rather miserable criteria. Actually, the use of a hedonic test to show a preference for food and medicine had been done by many researchers [43-45].

**CONCLUSION**

Based on the results of the printed mass and QC results, the printout of all formulas showed good results. From the three formulas, it can be concluded that secang wood extract (Caesalpinia sappan L.) can be made into ODT preparations that meet the requirements of Indonesian Pharmacopoeia, namely using HPC-H as a variation of concentration masking agent by 4%, 4.5%, and 5%. Based on the results of antioxidant testing using the DPPH method, the IC\(_{50} \) value of secang wood extract was 3.75 ppm, ODT extract of secang wood formula I was 3.173 ppm, ODT extract of secang wood formula II was 3.464 ppm, ODT extract of wood extract formula III was 3.614 ppm, and vitamin C as a comparison of 2.169 ppm. From the IC\(_{50} \) value, it can be concluded that the three formulas belong to the category of antioxidants which are very strong because they have an IC\(_{50} \) value of less than 50 ppm. The results of this study suggest that the ODT secang could be as an alternative to well-known antioxidants in the market including enzymes and other substances, such as vitamin C, vitamin E, and beta carotene.

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**AUTHORS CONTRIBUTIONS**

All the author have contributed equally

**CONFLICT OF INTERESTS**

There is no conflict of interest between authors.

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