Original article

Structure and Mechanical Properties of Rice Starch and Bovine Bone Powder Composite Films

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Abstract  Glycerol-plasticized starch/bovine bone nanopowder composite films were prepared from rice starch and six different compositions of bovine bone powders in aqueous suspensions (5, 10, 15, 20, 25 and 30 wt%) by casting, in order to study the effect of bovine bone nanopowders (BBN) on the rheology behavior of starch.  The aqueous suspension showed pseudoplastic fluid and near thixotropy behavior.  The BBN dispersion in films was analyzed by x-ray diffraction (XRD).  The morphology of starch nanocomposite films was studied by scanning electron microscopy.  The tensile test was performed on the nonocomposite film showing significant improvement in the tensile strength of up to 600% for the nanocomposite containing 15 wt% of BBN.  

Keywords: nanopowder, nanocomposite, rice starch, bovine bone powder

Natural bone is a nanocomposite consisting of calciumphosphate at 70 wt% and collagen at 20 wt%.  Calciumphosphate is in the form of calciumhydroxyapatite crystals in the size of nanometers and calciumphosphate base (? Matrix), which binds with collagen microfibrils.  A compound consisting of organic and inorganic materials has been developed in order to create structural and chemical properties that simulate natural bone.(1)

A hydroxyapatite and polymer compound mixture, which can be degraded biologically, has been developed to improve stiffness and tensile strength.  The compound mixture shows a reference point for use, and when the percentage of hydroxyapatite in the compound mixture is lower than the reference point, the compound mixture shows increased tensile strength as well as a suitable modulus for use in replacing natural bone.  In contrast, when the percentage of hydroxyapatite in the compound is higher than the reference point, a significant reduction in mechanical property is shown.  Recently, there have been many studies on compound that is degradable biologically.(1)

Polymer that has starch as one of its components was able to show a use in medicine, and since has been widely employed.  Starch is a polymer that can be degraded totally, and cheap when compared with other degradable compounds.  This compound mixture is compatible with the body, and has been tested both in vitro and in vivo.(2,3)
In this study, we used rice starch as original compound. For a longtime, rice has been the main food product of farmers in Thailand. In 2003, Thai export rice were the number one in the world, followed by India, Vietnam and the United States. In our world, the total production is 381.8 million tons of unmilled rice and 26.6 million tons of rice. Thailand can produce 26.1 million tons (number 6 of the world’s rice producing) countries of unmilled rice, and exports 6.6 million tons of rice (total cost 6.1 millions both). Normally, 1,000 kg of unmilled rice can produce 650 kg of rice, 100 kg of rice bran and 250 kg of chaff. In 2002, the main agricultural products exported from Thailand were rubber 36.7%, rice 35.2%, cassava 13.0%, vegetables (fresh, frozen and dried) 2.8% and others 11.3%. However, rice contributes 2.3% of the total export value. Therefore, there should be more research on rice to improve its value.

REVIEW ARTICLE

In 2001, Malafaya et al. successfully produced a mixture film from corn starch mixed with hydroxyapatite, which was obtained from chemical analysis. This mixture can be absorbed and degraded from the human body, as well as it having a reaction with human tissue. This property was proposed as having ability for use in humans. In 2007, Ruksudjarit, et al synthesized hydroxyapatite from bovine bone. They used a fresh bovine bone and removed the protein components by boiling, and burning at 800 °C. Then the dried bovine bone was ground and analyzed for its component by x-ray diffraction. It was found that the hydroxyapatite prepared from bovine bone had the same phase as purified hydroxyapatite.

Objective of reviewing articles

The review of articles was carried out in order to improve the basic knowledge of rice starch and bovine bone usage to produce a biomaterial that follows the pharmaceutical industrial standard. This material was then modified into a gel mixture using as a bone fixation for bone fractures in the human body or in other areas that do not require a weight load. In this study, a composite film was prepared from rice starch and hydroxyapatite obtained from bovine bone, with varying degrees of hydroxyapatite content. Furthermore, in order to prepare this film, the microstructure and mechanical property of this composite was also studied.

MATERIAL AND METHODS

Material

All material used and the preparation process of this composite followed pharmacopoeia, with no toxicity, no risk of malignancy development, and no cause of allergy and hypersensitivity reaction.

Bovine bone was prepared according to the American Society for Testing and Materials (ASTM); designation F1581-99, 2000. That is to say, the compound did not contain organic materials. The concentration of trace elements had to be as follows: arsenic < 3 ppm, cadmium < 5 ppm, mercury < 5 ppm, lead < 30 ppm, and overall heavy metals < 50 ppm. Then the particle of bovine bone was reduced in size by high speed grinding for 3 hours.

The last property of the composite had to be insoluble in water, absorbable in the human body, and able to repair bone. The rate of bone reparation had to compatible with the rate of degradation of the composite. Additionally, this composite must be completely degraded in the human body.

Methods

Purified rice starch (Era-Tab® - Erawan Pharmaceutical Research Laboratory Thailand) at 3 gm was dissolved in 100 mL of distilled water, heated to 85 °C (the optimal gelatinization of rich starch occurs at 75-85 °C), and the mixture was stirred for 20 min to produce a clear semi-liquid solution. Then, 1 gm of gelatin (Fluka, Germany) was mixed in 3 mL of polyvinylalcohol (PVA), and added to 3 mL of glycerol (BP, USP, China). This solution was dissolved in 50 mL of distilled water at 65 °C, by stirring for 15 min. The starch solution was mixed with the gelatin solution, and then the bovine bone powder was added at 0, 5, 10, 15, 20, 25 and 30% by weight. The solution was stirred for 30 min until the bovine bone powder mixed well with the rice starch solution.
**Laboratory testing**

The following tests were performed:

1. Determination of the microstructure of the surface of the hydroxyapatite particle and composite films using scanning electron microscopy (SEM).

2. Analysis of the viscosity of the suspension behavior using the Brookfield Viscometer Model DV-VIII+.

3. Confirmation of the inorganic component of the hydroxyapatite prepared from bovine bone using x-ray diffraction.

4. Test for tensile strength using the Universal Machine.

**RESULTS**

After XRD tests, the bovine bone powder was confirmed as having a crystal similar to hydroxyapatite crystals (Fig. 1).

When the bovine bone powder was determined by SEM, an agglomerate of nanometer hydroxyapatite from bovine bone was seen (Fig. 2). In 2000, Tomas Raming explained the reason why nanoparticle hydroxyapatite formed an agglomerate of nanoparticles that had a van der Waal force, when compared with other forces acting on these particles. The more van der Waal force increased, the more the nanoparticles came closer together, resulting in higher chemical bonds between these nanoparticles. Although there was no chemical bonding, the increase in van der Waal force made these nanoparticles become closer together. This process is called “Aggregation”. However, in Figure 1, although there is an agglomerate of hydroxyapatite the individual grain size is less than 100 nm.

Table 1 and Figure 4 show that the composite with 15wt% had the highest tensile strength, and was higher than the mixture which did not contain bovine bone of up to 600%. Furthermore, the broken surface characteristic from tension showed the distribution of bovine bone powder on the composite film (Fig. 5), making a good adhesion force between the surface of bovine bone powder and...
Figure 3a and 3b. Graph showing flowing behavior of supernatant when bovine power was added at 0, 5, 10, 15, 20 and 25wt%.

**Table 1.** Mechanical property of the compound mixture of rice starch and bovine bone powder after 2 hours gliding.

<table>
<thead>
<tr>
<th>Quantity HA (% wt)</th>
<th>Youngsmodulus (MPa)</th>
<th>Tensile strength (MPa)</th>
<th>Strain at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10.43±0.50</td>
<td>2.19±0.07</td>
<td>112.49±2.35</td>
</tr>
<tr>
<td>5</td>
<td>106.96±12.79</td>
<td>10.01±0.33</td>
<td>95.29±2.80</td>
</tr>
<tr>
<td>10</td>
<td>127.07±4.17</td>
<td>11.76±0.49</td>
<td>84.66±3.49</td>
</tr>
<tr>
<td>15</td>
<td>172.81±6.23</td>
<td>13.70±0.45</td>
<td>62.51±1.96</td>
</tr>
<tr>
<td>20</td>
<td>144.47±7.96</td>
<td>12.24±0.40</td>
<td>65.09±3.81</td>
</tr>
<tr>
<td>25</td>
<td>137.10±7.30</td>
<td>11.99±0.32</td>
<td>75.69±2.89</td>
</tr>
<tr>
<td>30</td>
<td>141.35±13.03</td>
<td>12.09±0.33</td>
<td>72.22±3.39</td>
</tr>
</tbody>
</table>

Figure 3a and 3b show that when 15%wt of bovine power was added to the starch mixture, the composite had the lowest viscosity and the flowing behavior was close to thixotropic.

**DICUSSION**

This study, showed that a mixture of rice starch film without bovine power had the lowest tensile strength, and highest viscosity, leading to a poor flowing property. This resulted in a poor mold formation, as the force that made the suspension was inconsistent, and the development thixotropic. However, when the bovine bone powder was added at 15%wt, it made the distribution of the bovine starch powder uniform in the rice starch film, causing less thixotropic and pseudoplastic flowing. When making a mold, this 15wt% composition mixture had the highest tensile strength.

![Graph showing tensile strength when various percentages of bovine bone powder were added.](image)
because the force was evenly distributed through the bovine bone powder to the rice starch film. Additionally, as the nanoparticle had the higher surface, the surface tension was also increased, making a good adhesive force between the bovine bone powder and rice starch.

When more than 15%wt of bovine bone powder was added, the tensile strength decreased. This could be explained in that the bovine bone powder aggregated into a large clump, which interfered with the flow of suspension, thus making an uneven distribution of nanoparticles, and leading to decreased tensile strength. However, there was also the effect of nanoparticles in the tensile strength of rice starch. At 20 and 25%wt, there was not much difference between the tensile and highest tensile strength. Weves the less 30%wt, the nanoparticles tended to be a large powder particle, which caused a problem in forming a mold.

ACKNOWLEDGEMENT

We thanks the National Research Council, the Faculty of Science and the Graduate School of Chiang Mai University, and the Office of Higher Education Commission.

REFERENCES


Figure 5. Scanning electron microscopy showing a broken surface of the compound mixture of rice starch and bovine bone powder at 15%wt
โครงสร้างและสมบัติกลของฟิล์มวัสดุผสมแป้งข้าวเจ้ากับผงกระดูกวัว

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ผงแป้งข้าวเจ้าและผงกระดูกวัวส่วนผสมเตรียมโดยใช้กลีเซอรอลเป็นพลาสติก-ไซเซอร์แล้วเติมผงนาโนกระดูกวัวในปริมาณต่างกัน 6 ค่าคือ 5, 10, 15, 20, 25 และ 30 เปอร์เซ็นต์โดยน้ำหนัก เพื่อศึกษาพฤติกรรมการไหลของสารแขวนลอยที่มีน้ำเป็นตัวทำละลายและหาผลของผงกระดูกวัวต่อสมบัติเชิงกลของฟิล์ม แล้วทำการลักษณะและสภาพของผิวงานด้วยวิธีการเลี้ยวเบนของรังสีเอกซ์ และวิเคราะห์เพดานด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด และจากการขึ้นรูปได้ผลการพิจารณาพบว่าสารแขวนลอยมีพฤติกรรมการไหลที่เป็นแบบซูโดพลาสติก มีโฮโมทรอปิคต่ำ และฟิล์มวัสดุผสมเข้ากันได้ที่ร้อยละ 15 เชื่อมต่อกับผิวผิว 2553;49(4):139-144.

คำสำคัญ: ผงแป้ง, ผงกระดูกวัว, ฟิล์มวัสดุผสมนาโน

คำอ้างอิง: ผงแป้ง, ผงกระดูก, ฟิล์มวัสดุผสมนาโน, ฟิล์มวัสดุผสมแป้งข้าวเจ้ากับผงกระดูกวัว