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Research on Mechanical–Structural and Oil Yield Properties during *Xanthoceras sorbifolium* Seed Oil Extraction

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Abstract: Products from *Xanthoceras sorbifolium* Bunge seed have gained extensive attention for various applications, especially in the fields of edible oils and industrial applications. In order to study seed kernel mechanical–structural behavior and oil yield mechanisms during extrusion, we set up a self-developed texture analyzer with in situ microscope observation. Test results indicated that seed kernel oil yield and pressing energy showed an approximately parabolic shape under pressing strain, and maximum oil yield reached 25.7%. Only local tissue damage occurred on seed kernels at strain 45–85%, cracks formed from the kernel edge to the inside zone and small cracks obviously increased in number, corresponding with the oil yield and energy–strain curve. The effect of speed on oil yield showed an opposite trend to strain effect; high pressing speed led to lower oil yield due to the short time for oil precipitation and lower pressing energy. Dwell time obviously promoted oil output within 600 s. Drying temperature had a negative effect due to structural change. Oil yield was almost zero at temperatures below 120 °C. The oil yield and pressing energy relation curve was obtained by polynomial fitting; optimal seed kernel oil pressing conditions were strain 95%, 0.1 mm/s, 20 °C, dwell time 600 s. The research provides in-depth theoretical guidance for *Xanthoceras sorbifolium* Bunge oil production.

Keywords: mechanical–structural property; oil yield; in-situ observation; extraction; *Xanthoceras sorbifolium* Bunge seed



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1. Introduction

The indigenous shrub *Xanthoceras sorbifolium* Bunge (yellow horn), belonging to the Sapindaceae family, is native to northern China [1–3]. It has great survival ability [4,5]. Its seed consists of a seed kernel and a hard outer shell. *X. sorbifolium* seed oil content within the shell is 30–50%, and the seed kernel has a higher oil content of 55–65% [6]. It is a good source of unsaturated fatty acids (85–93%) and is rich in linoleic and oleic acids. It can be used in the field of edible oils, and *X. sorbifolium* has a high nutritional value and various medicinal applications [7–9]. *X. sorbifolium* oil is an edible vegetable oil. It has been widely used in the biodiesel industry as a sustainable bioenergy source [6,10], in industrial lubrication oil applications, etc.

Vegetable oil and industrial applications require seed oil extraction. Vegetable oil is generally extracted from oilseeds by expeller pressing and solvent extraction [11–13]. Solvent extraction is more costly and less pure than mechanical pressing; the mechanical method is generally used in industrial extraction, which mainly involves continuous pressing within decreasing space [14]. Seed oil yield is an important index in the oil extraction process, and is mainly affected by moisture content, temperature, load and rate etc. [15–18]. Oil extraction processes have attracted much research attention. Studies have sometimes focused on direct factors affecting oil yield, such as load, moisture content, temperature,

speed, etc. For instance, Evon and Tambunan studied the effect of temperature, rate, and preheating time on the oil yield of *Jatropha curcas* seeds using a mechanical extraction method, and physical properties were examined [19,20]. Fadhlullah studied the effect of different moisture content and seed size on *C. inophyllum* oil extraction using a screw press and carried out oil characterization [21]. In compression tests on almonds, Martinez obtained the highest oil recovery conditions under different moisture contents and pressing temperatures [22]. The mechanical properties of different plant seeds are different during screw pressing. Research on various oilseed crops can contribute to the development of screw-pressing processing [23–26]. Some previous research also focused on the mechanical properties of oilseeds. Deformation and energy are other important indicators characterizing the mechanical properties of seeds. Herák and Kabutey investigated the energy demands of *Jatropha curcas* seeds for oil extraction under working conditions, and established a mechanical model for oil yield and deformation [27,28]. In addition, the seed oil yield of *Xanthoceras sorbifolium* Bunge under different extraction methods (subcritical n-butane, supercritical carbon dioxide (CO₂), and Soxhlet extraction) has also been studied, and the basic properties of seed oil and optimal oil extraction methods have been compared [1]. Some oilseed needs to be dehulled before pressing extraction, and the related mechanical studies are useful. Yang and Chen studied the fracture mechanism and shell-breaking behavior of castor seeds during the hulling process by the in-situ observation with a self-made texture analyzer [29]. Noraphaiphipaksa [30] demonstrated that it is fundamental and important to study the mechanisms of oil yield and shell breaking.

Xanthoceras sorbifolium Bunge is of great value for exploitation and utilization. Few studies on the mechanical properties and oil extraction of *X. sorbifolium* have been conducted. Most of the previous research has focused on seed oil extraction, but single seed grain mechanical–structural damage properties and oil outflow are fundamental. In this paper, compression tests of single seed grain were run under different conditions using a self-developed texture analyzer with in situ optical observation. The mechanical–structural damage behavior and oil production characteristics were analyzed in depth. Models of mechanical–structural changes and oil yield were developed.

2. Materials and Methods

2.1. Materials and Sample Preparation

Xanthoceras sorbifolium Bunge seeds were obtained from Shanxi Province, China. Initial properties such as oil content and ash content of the seed were measured according to standard methods [31–34]. The initial properties of *Xanthoceras sorbifolium* Bunge seeds are shown in Table 1. To ensure higher consistency of test results, all seeds were placed under the same laboratory conditions at 20 °C. Seeds were hand-hulled and seed kernels of similar size and thickness 8.0–8.3 mm were screened with an optical microscope. To prepare samples under dry temperature conditions, seed kernels were exposed to drying in a heating oven (Beijing Yongguangming Medical Instrument Co., Ltd., Beijing, China) for 30 min.

Table 1. The basic properties of *Xanthoceras sorbifolium* Bunge seed.

No.	Item	Relative Content (%)
1	Seed oil content	30~40
2	Seed kernel oil content	55~65
3	Seed kernel moisture	≈4
4	Seed kernel weight/seed weight	40~50
5	Unsaturated fatty acids	85~93
6	Saturated fatty acids	≤10
7	Moisture and volatile content in oil	≈0.4
8	Ash	≈2.3

Table 2. Factors and levels of the compression tests.

Levels	Factors			
	Strain (Deformation Degree) during Test (%)	Pressing Speed (mm/s)	Dwell Time (s)	Temperature (°C)
1	45	0.1	120	20
2	55	0.5	240	60
3	65	1.0	360	90
4	75	1.5	480	120
5	85	2.0	600	150
6	95	—	720	—

To obtain reliable data, the seed was kept in the same orientation for each test, and each test was repeated at least five times. Seeds were weighed before and after each test. Probe movement speed was set to 2.0 mm/s before contact and after compression. Probe sensing force was 0.2 N during the shell-breaking test, and 0.1 N during oil pressing. The influence of press holding time on kernel oil yield was considered.

2.2.2. Structural–Mechanical and Oil Output Characterization

Fracture property, which includes rupture force and energy, is one of the seed's mechanical–structural properties [35]. Structural damage and rupture are followed by oil outflow; rupture is characterized by a rapid decrease in compression force when structural fracturing occurs. Rupture energy can be described as the energy required for structural rupture (contact failure); its mathematical calculation is obtained by integrating the force–deformation curve before the fracture point, as shown in Equation (1) [36,37].

$$E = \int_0^x Fd\delta \quad (1)$$

where E is the working energy (N·mm), F is rupture force (N) and δ is structural deformation (mm).

To study in depth the oil output characteristics of oil seeds, seed strain (deformation degree) can be used as an independent variable indicating the degree of structural damage, with oil yield as the target value. The strain and oil yield are calculated as shown in Equations (2) and (3).

$$S = \frac{\Delta L}{L} * 100\% \quad (2)$$

where S is strain, ΔL denotes the deformation of the seed kernel under compression and L is seed kernel thickness.

$$Y = \frac{\Delta m}{m} * 100\% \quad (3)$$

where Y is oil yield, Δm indicates the weight loss of the seed kernel (approximately equal to the extracted oil volume) before and after compression testing and m is seed kernel initial weight.

3. Results and Discussion

3.1. Compression Curve Analysis for *X. sorbifolium* Seed

Seed Kernel Structural–Mechanical Properties and Oil Output during Extrusion

The mechanical–structural behaviors of *X. sorbifolium* seed can be characterized by pressing force, energy and deformation. Referring to the force curve in [38], Figure 2 shows the force–time and force–displacement relationships of *X. sorbifolium* seed kernel deformation at degree 85%, speed 0.1 mm/s and dwell time 240 s. The two curves are correspondent. The shaded area was integrated using Equation (1) to obtain seed kernel pressing energy. During the pressing stage of curve 0-a or 0-a*, the seed kernel showed elastic deformation and force increased linearly, while strain was small. Observing the force–

displacement curve, compression force reached the critical point of plastic deformation at point 'a'. Continuous compression work produced obvious structural damage (cracks) in the seed kernel. Point 'a' can be described as the structural fracture damage point of the seed kernel at 58 N. Due to the seed kernel's soft texture, rupture force decay during this fracture damage was relatively gentle. Structural damage imaging of point s-s* in Figure 2 shows the structural damage form, which occurred under displacement at 2.3–3.1 mm (time 4.6–6.2 s). The second half of the force–displacement curve shows a rapid force increase during continuous pressing. Peak force reached 267.9 N at point 'b'. During this period, structural damage was further enhanced, the number of cracks generated obviously increased and no initial crack damage fluctuation force curve shape occurred as in region 's'. The force–time curve indicates the effect of dwell time on peak force, which cannot be characterized by the force–displacement curve. As the seed kernel entered the pressing dwell period after achieving the final displacement distance, the force–time curve shows a rapid decay of peak force, and then a slow decrease to point 'c' with 167.4 N. Seed kernel dwelling pressing can further intensify the internal structural damage, leading to further oil precipitation from the internal oil structure. Higher pressure brings more significant oil percolation [39]. As oil decreases and kernel morphology expands, a small decrease in internal pressure and external load occurs.

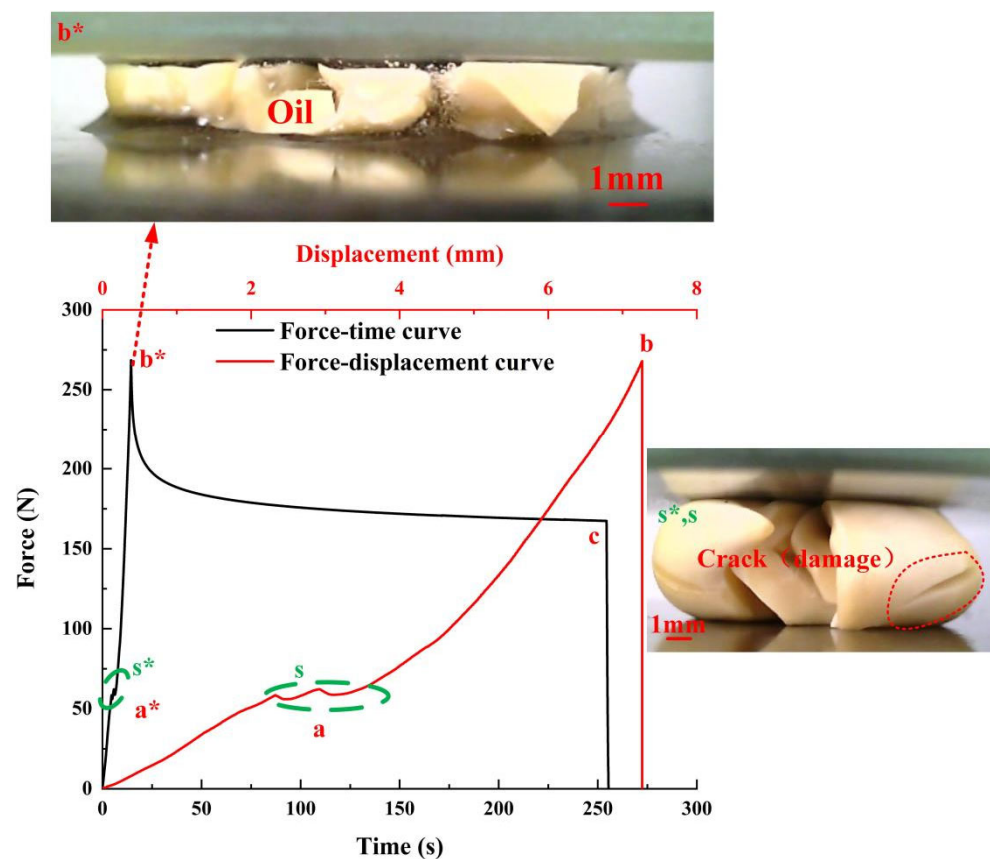


Figure 2. The force–time and force–displacement curves for seed kernel oil extraction.

3.2. Effect of Pressing Strain on the Mechanical–Structural Properties and Oil Yield of Seed Kernels

Compression displacement directly leads to seed kernel deformation of different degrees (strain), which is a fundamental point in the study of the mechanical–structural properties and oil output of seed kernels. Figure 3 shows seed kernel oil yield and pressing energy versus displacement under conditions of pressing speed 0.1 mm/s, dwell time 240 s and laboratory temperature 20 °C. The oil yield–strain and energy–strain relation curves

are shown in Figure 3a,b; the oil yield and energy curves are correlated and each shows an upward parabola-like shape.

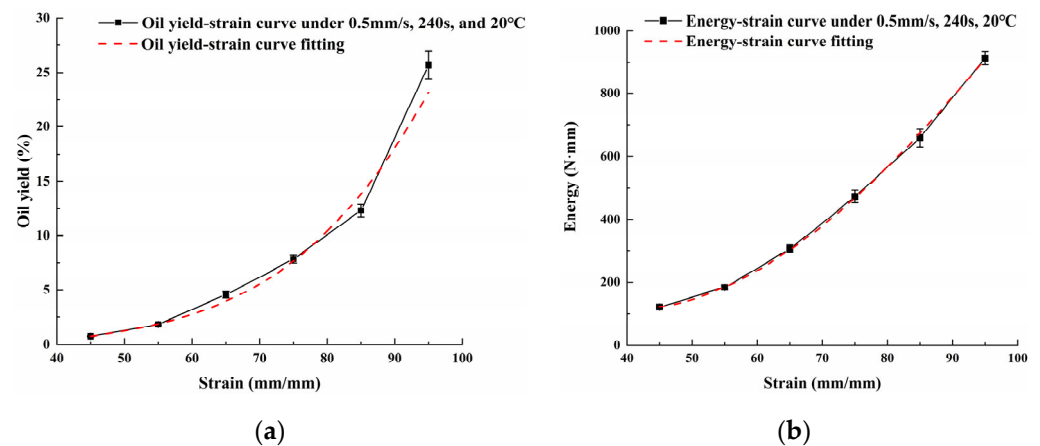


Figure 3. Seed kernel oil yield–strain curve (a) and energy–strain curve (b) under conditions of 0.5 mm/s, 240 s and 20 °C.

Overall, the growth trend of oil yield showed a slow and then sharp increase. The increase of oil yield was significant at strains greater than 65%. However, the energy showed a steady increase with strain growth. Under a strain of 45%, oil yield was close to 0; meanwhile, the pressing energy was 122 N·mm, which caused elastic deformation and cracking at the outer edges. With strain increasing to 95%, more pressing energy was created, and the seed kernel damage was intensified, leading to more oil precipitation. The corresponding in situ observation image of seed kernel structural damage is shown in Figure 4. The oil yield was close to 0 under an extrusion strain of 45%, with no obvious damage presented. The unique structure and soft material of the *X. sorbifolium* seed kernel give it strong load-bearing capacity and crack resistance. During the pressing period, the seed kernel was still mainly in the elastic deformation stage and little tissue damage occurred, as shown in Figure 4a. The only cracks appeared at outer edge positions on the kernel main body, and no kernel root part damage happened. As extrusion strain increased, the number of seed cracks obviously increased; existing seed kernel damage also intensified, and the oil outflow was mainly from the damaged part. With increasing strain, more cracks were formed, as shown in Figure 4a–f. The oil yield–strain curve showed an upper parabolic trend, corresponding with the crack damage degree, as seed kernel oil yield increased rapidly. The most significant oil yield was achieved at strains of 85–95%, reaching a maximum of 25.7%. As can be seen from Figure 4, greater strain led to more severe mechanical damage to the kernel. Seed kernels showed the greatest overall damage at strains of 85–95%. The damage at 45–75% strain was not dissimilar, but was less extensive. The damaged part gradually extended to the internal and central parts of the kernel main body. Usually, mechanical damage extends with increasing load to a higher degree of strain [40,41]. Maximum damage at a strain of 85–95% can be seen in Figure 4. Oil output was most significant at this period. The process of damage extension occurred at 45–75% strain, where oil precipitation was limited.

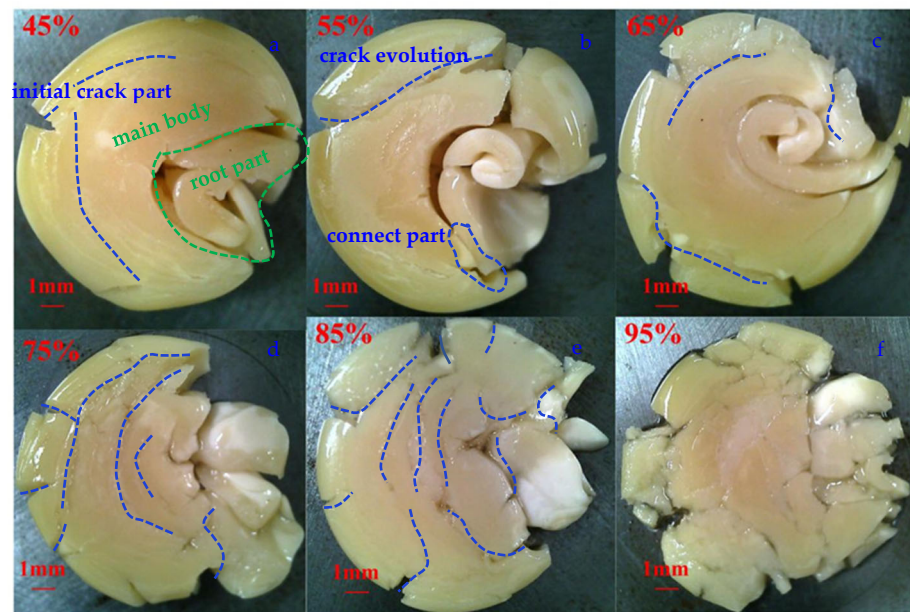


Figure 4. In situ observation of seed kernel structural damage under different strains: (a) 45%; (b) 55%; (c) 65%; (d) 75%; (e) 85%; (f) 95%.

Based on the relevant literature [42], polynomial fitting of strain curves under different target factors was performed and analyzed. The fitted expression of oil yield–strain is shown in Equation (4). The fitted energy–strain expression is shown in Equation (5).

$$y_1 = 0.7772x - 0.0161x^2 - 0.0001x^3 - 13.0994 \quad (4)$$

$$y_2 = -33.7175x + 0.5016x^2 - 0.0014x^3 + 746.5779 \quad (5)$$

where x represents the strain ($x \in (45,100)$), y_1 represents oil yield from a seed kernel, $R^2(y_1) = 0.9886$, y_2 represents energy and $R^2(y_2) = 0.9998$.

3.3. Effect of Pressing Speed on Mechanical–Structural Properties and Oil Yield of Seed Kernels

Velocity is a fundamental parameter of mechanical properties affecting the energy requirement for seed damage and oil extraction. Compression oil extraction testing of seed kernels was carried out under different pressing velocities at strain 85%, 240 s and 20 °C. Figure 5 shows the oil yield–strain and energy–strain relation curves. The maximum seed kernel oil yield was about 16.95% at a speed of 0.1 mm/s. Oil yield gradually decreased as speed increased, and the decrease rate gradually became slower. Minimal oil yield was 8.9% at 2.0 mm/s. Lower speed can prolong the interaction time during seed compression [43,44], so oil precipitation is higher, leading to increased oil yield.

To consider another point, although higher pressing speed led to larger fractures, small tissue damage was reduced (Figure 6), which led to less oil output from oil cells, explaining the observed oil precipitation. Meanwhile, from the energy–speed curve shown in Figure 5b it can be seen that the pressing energy decreased with increasing speed. Energy was almost stable at 563 N·mm when reaching a speed of 1.0 mm/s, which confirms the oil extraction decreased with increasing speed. Speed had a significant negative effect on the oil yield and energy of seeds and kernels. This negative effect tended to stabilize when the speed was higher. Therefore, a lower squeezing speed should be set appropriately to improve the oil yield.

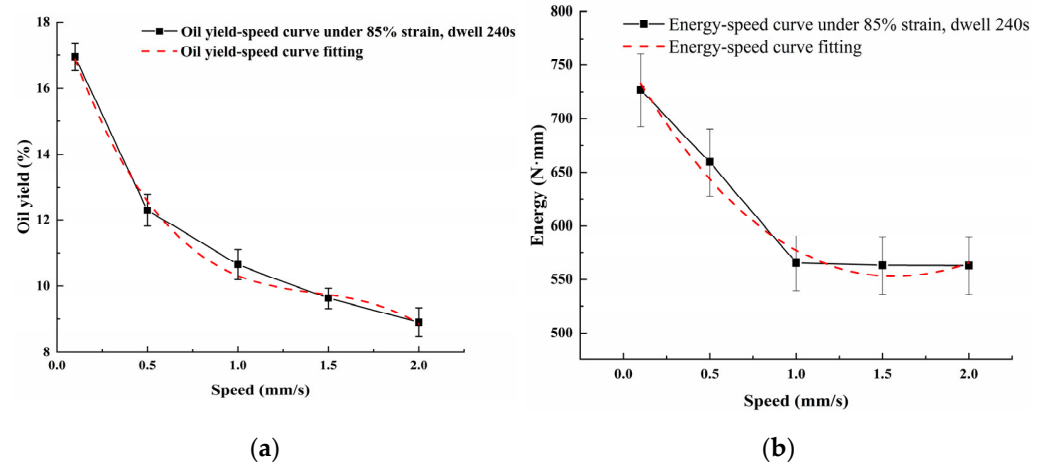


Figure 5. The kernel oil yield-speed curve (a) and energy-speed curve (b) at 85% strain, dwell time 240 s, 20 °C.

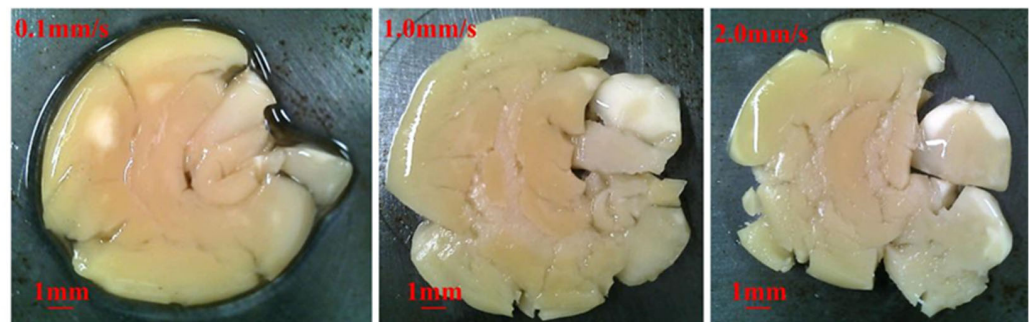


Figure 6. In situ damage observation of seed kernel at 0.1 mm/s, 1.0 mm/s and 2.0 mm/s.

The oil yield–speed and energy–speed relationships were fitted by polynomial functions, as shown in Figure 5a,b. The fitting expressions are as follows:

$$y_6 = -16.6248x + 11.0963x^2 - 2.5903x^3 + 18.4394 \quad (6)$$

$$y_7 = -287.3255x + 113.4416x^2 - 9.2651x^3 + 760.2172 \quad (7)$$

where x represents the speed, y_6 represents the oil yield, $R^2(y_6) = 0.9958$, y_7 represents energy and $R^2(y_7) = 0.9689$.

3.4. Effect of Dwell Time on Oil Yield of Seed Kernels

Figure 7 shows the effect of dwell time on seed kernel oil yield at 85% strain, 0.5 mm/s and 20 °C. Figure 7b shows the pressing energy at different dwell times; the energy was changed slightly, at around 550 N·mm, without obvious influence. Dwell time had a significant effect on the improvement of oil yield. The oil yield of seed kernels was the lowest at a dwell time of 120 s, reaching about 12%. At dwell times between 120 s and 360 s, oil yield increased slowly. Between 360 s and 600 s, oil yield increased significantly. At dwell times exceeding 600 s, the growth rate of oil yield decreased and stabilized at about 20%. Darcy's seepage law shows that it takes some time for oil to seep out from a crevice to outside, and the longer the time, the more oil will flow out [45,46]. When dwell time is too long, oil and internal pressure decrease so that oil extraction rate gradually tends to level off. Oil extraction efficiency will be reduced if time is over-extended. Considering economic issues and production efficiency, optimal oil yield was highest at 19.3% at a dwell time of 600 s.

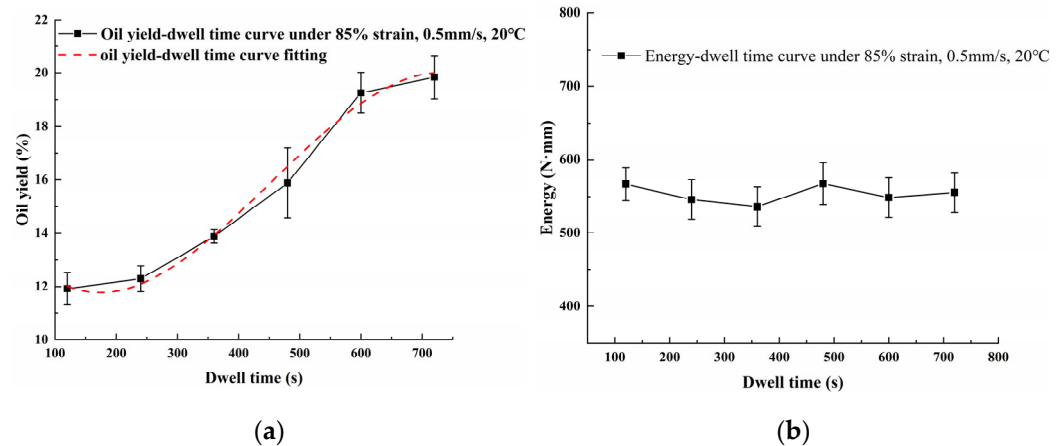


Figure 7. The kernel oil yield-dwell time(a) and energy-dwell time curve (b) under strain 85%, 0.5 mm/s, 20 °C.

The relationship between oil yield and dwell time was fitted using a polynomial function. The fitting expression is as follows:

$$y_8 = -16.6248x + 11.0963x^2 - 2.5903x^3 + 18.4394 \quad (8)$$

where x represents the dwell time, y_8 represents oil yield from a seed kernel and $R^2(y_8) = 0.9958$.

3.5. Effect of Drying Temperature on the Mechanical–Structural Properties and Oil Yield of Seed Kernels

The drying process can affect the moisture content of the seed, and may change the texture. The drying weight loss rate is the ratio of seed kernel weight loss after drying treatment to the initial mass. Figure 8 shows oil yield and drying weight loss rate curves under 85% strain, 0.5 mm/s and 240 s. The seed kernel pressing damage in situ observation images are also shown in Figure 9. The drying weight loss curve shows that weight loss rate increased as the temperature increased from 20 °C to 120 °C, with the trend slowing at temperatures above 120 °C. The maximum weight loss rate was 5.3% at 150 °C. There was a negative correlation between oil yield and drying temperature. From the oil yield–drying temperature curve, it can be seen that the highest oil yield of seed kernels dried at 20 °C was about 12%. Upon increasing the drying temperature, the oil yield decreased significantly. Notably, increasing the temperature to 150 °C after 120 °C led to higher oil yield. Oil yield at the special temperature point 120 °C was 0.53%. Figure 8b shows that the pressing energy decreased as the temperature increased from 20 °C to 120 °C. When temperature increased from 120 °C to 150 °C, the pressing energy increased, which corresponded with the oil yield rise at 150 °C. Drying causes substantial water loss, increases the oil viscosity and makes it more difficult for oil to seep out of the oil cells [47]; therefore, the oil yield decreases significantly. Additionally, as can be observed from Figure 9 that at temperatures above 90 °C the seed kernel broke into large blocks, leading to less oil output on the surface. At 150 °C, the seed surface showed an apparent brownish-yellow color, indicating a possible change in texture that made tissues less tough and more susceptible to damage, thus causing the improvement of oil yield. The higher yield might be due to the decomposition of other components (not the oil). Drying temperature and pressing energy can reduce the moisture content of seed. As water content decreases, the hardness of the seed increases. Proper drying makes the seed fiber composition tougher [48]. However, detailed reasons for this need to be supported by our future work.

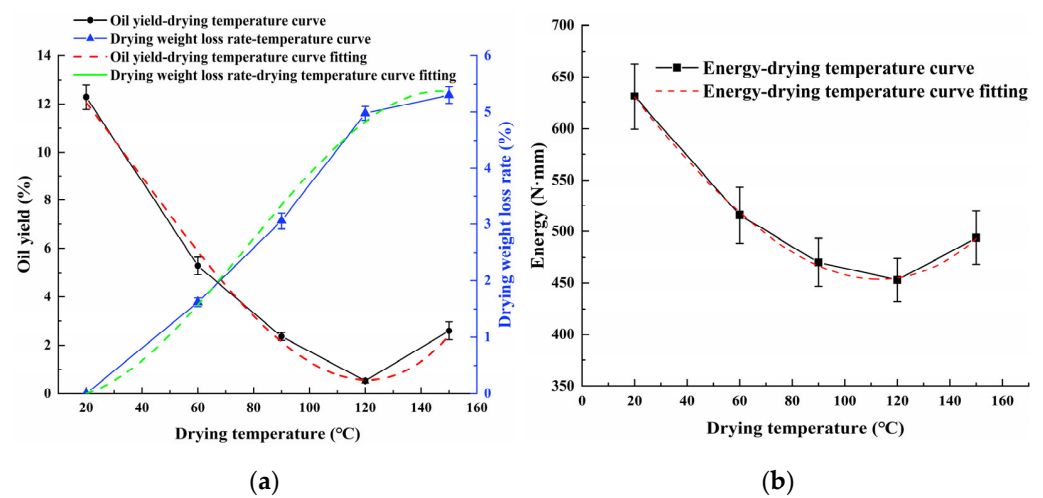


Figure 8. The oil yield–drying temperature curve(a) and energy–drying temperature curve (b) under 85% strain, 0.5 mm/s, 240 s.

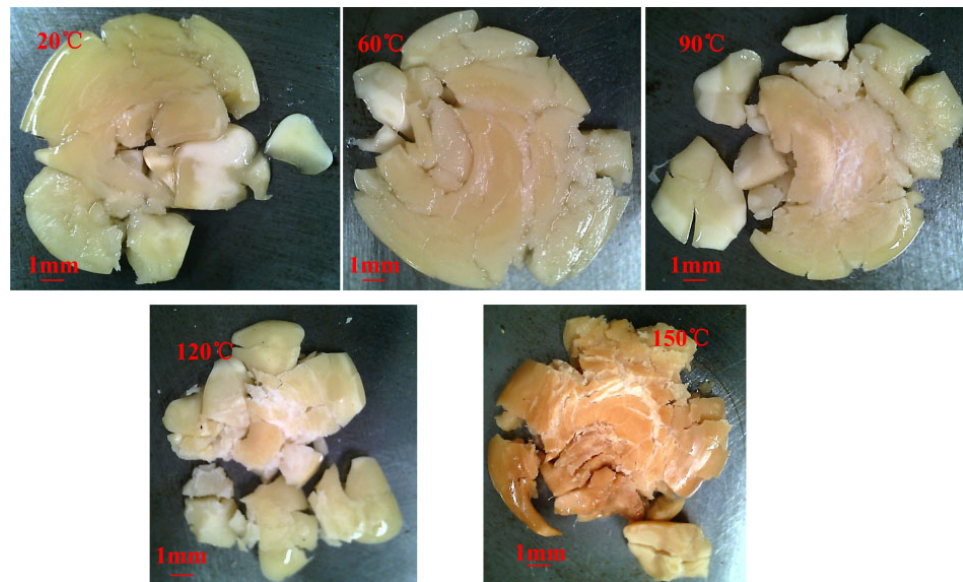


Figure 9. In situ observation of seed kernels under different drying temperature conditions.

The relationships between oil yield (drying weight loss rate, energy) and drying temperature were fitted using polynomial functions. The fitting expressions are as follows:

$$y_9 = -0.1246x + 0.0009x^2 - 7.6861 \times 10^{-6}x^3 + 14.8449 \quad (9)$$

$$y_{10} = -0.0194x + 0.001x^2 - 4.313 \times 10^{-6}x^3 - 0.0181 \quad (10)$$

$$y_{11} = -3.4533x + 0.0038x^2 - 6.74 \times 10^{-5}x^3 + 697.418 \quad (11)$$

where x represents the drying temperature, y_9 represents oil yield on a seed kernel, $R^2(y_9) = 0.9926$, y_{10} represents drying weight loss rate, $R^2(y_{10}) = 0.9985$, y_{11} represents energy and $R^2(y_{11}) = 0.9985$.

4. Conclusions

In this study, mechanical–structural behavior and oil-yield mechanisms of *X. sorbifolium* seed kernels were investigated in relation to pressing strain, speed, dwell time and drying temperature. Seed damage was characterized with in situ observation. The trend

towards increasing oil yield and pressing energy under large displacement was obvious. It was observed that fracture cracking extended from contact area to seed tip. Oil yield under strain presented an approximately parabolic shape. Oil yield was 25.7% under a strain of 95%. Seed kernels were almost fully damaged under strains of 85–95%, with the most significant oil production occurring under these conditions. Pressing energy increased linearly with pressing speed, and oil yield decreased gradually. Other conditions being constant, the maximum oil output was about 17% at the lowest compression speed (0.1 mm/s). Dwell time can promote oil precipitation, but working effect diminished when dwell time was too long. The best oil output rate occurred at a dwell time of 600 s. Drying changes seed hardness by changing moisture content. Pressing energy decreased with the increase in drying temperature, and oil yield was negatively affected by drying. When temperatures was increased to 150 °C, oil yield increased, corresponding with the pressing energy–temperature relation. This result was related to tissue structure changes caused by drying temperature, which could be easily observed in situ.

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