

Agglomeration during reactive extrusion of particle-based biomass pellets

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Abstract. Creating mechanically sound aggregates from powder or grain-like feedstocks is a fundamental challenge across industries. One example is the production of pellets from organic materials, like wood residues and cereal grains, for biofuel and animal feed applications. However, the handling and processing of these organic powders is complex, and the binding mechanisms between particles remain poorly understood at physicochemical level. In this work, we address this knowledge gap by analyzing the energy consumption of physicochemical binding mechanisms during reactive extrusion of biomass pellets, by conducting experiments in an industrially relevant pilot plant. Understanding the mechanisms that drive this agglomeration process under various conditions and within various application domains are therefore important subjects of research into granular material dynamics.

We focus on the heat flow into the organic ingredients during the agglomeration process induced by *reactive extrusion* and find that not all the heat increases the product temperature. Instead, some heat is absorbed by internal changes such as phase transitions or chemical reactions. We identify that the energy absorption mechanism changes significantly once the material reaches the so-called stickiness temperature (T^*). Below T^* , the ingredient temperature increases linearly with the energy input and pellets do not bind well; above T^* , the material absorbs energy to undergo transformations that greatly improve the agglomerate strength.

Our findings indicate how further work can probe agglomeration kinetics in more detail and show that industrial processes can fine-tune product quality by controlling heat flow during steam conditioning and extrusion. We tested this framework with various ingredient mixtures and confirmed its robustness despite chemical differences. Our results highlight the importance of ingredient-specific physicochemical properties in reactive extrusion and open new directions for optimizing particle agglomeration processes.

1 Introduction

Many powder-like or granular materials gain enhanced functionality only after agglomeration—where individual particles are consolidated into a cohesive structure. This transformation enables a wide range of applications: powdered clay becomes ceramic tiles, flour becomes bread, and dry minerals become construction bricks. In each case, consolidation promotes contact between neighboring particles, allowing for interfacial interactions that drive mechanical strength, transport properties, or chemical stability.

One increasingly relevant agglomeration technique is reactive extrusion, in which chemical modification, structuring, and shaping occur simultaneously within a single continuous process [1, 2]. In such systems, mechanical energy is supplied by extruder screws, but local chemical reactions—such as polymerization, cross-linking, or phase transitions—may provide additional energy input or

lead to structural transformations [3]. One example of a physicochemical alteration relevant in this context is sintering, where heat promotes bonding at particle interfaces through diffusion or solid-state reactions. Ultimately, the formation of bonds at particle contacts determines the mechanical integrity and quality of the final agglomerated product, often quantified by metrics such as hardness or durability[3].

One particularly interesting agglomeration process is biomass pelleting. The pelleting process is a hydrothermal process in which finely ground powders or particulate solids are processed within a pellet mill, to produce dense cylindrical pellets. These pellets have diverse applications in areas such as compound animal feed [4], but also bio-fuels [5], and recently, (co-)pelletizing municipal solid waste [6]. By improving the bulk density of powders, pelleting enhances material handling and transportation and creates a higher value end product for specific markets. However, despite its industrial relevance, the underlying mechanisms that enable the formation of durable pellets remain poorly understood. This raises a fundamental question: “which mechanisms enable the formation of

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inter-particle bonds, transforming loose ingredients into durable agglomerates?"

In our previous works [7, 8], we introduced pelleting as a lubrication-sensitive process in which the concept of an ingredient-specific temperature, T^* , referred to as the *stickiness temperature* determines the mechanical fate of the pellet. Below T^* , agglomeration does not occur, whereas at process temperatures above T^* , ingredients consolidate to form high quality pellets. In Benders *et al.* [8], we showed that physical pellet quality data (PDI), an industrial metric for the pellet's mechanical stability, obtained under varying conditions, collapsed onto a single curve when plotted against pellet temperature measured after the die. This collapse highlights the critical role of achieving an ingredient-specific processing temperature during pelleting, outweighing other parameters such as production rate, in producing high-quality pellets. However, the underlying reason for this collapse remained unclear. Here, we extend our analysis by evaluating the energy exchange between the production process and the processed materials within the pellet mill, to explain why ingredients bond once process temperatures exceed the mixture's stickiness temperature, T^* .

In this work, we show that successful agglomeration of the loose ingredients that e.g. serve as the diet for animals, such as ground corn and soy, into durable pellets requires not only mechanical forces but also explicit energy absorption by the ingredients to form lasting, "sintered" contacts. Based on our experimental data, we propose that exceeding T^* induces significant energy uptake by the ingredients, due to physicochemical transitions. These include molecular-level changes such as phase and state transitions (e.g., glass transitions, melting), as well as chemical changes (e.g., protein denaturation). Such transformations enable extruded particles to shift from stiff and non-sticky to soft and adhesive, thus facilitating pellet formation [9, 10]. We collectively refer to these energy-absorbing phenomena as physicochemical processes, emphasizing their central role in initiating inter-particle bonding during pellet manufacturing.

Finally, this work positions pellet manufacturing as a model system for the reactive extrusion of organic materials. This concept, well-established in the plastics industry [11, 12], holds promise for novel applications in food and biomass processing.

2 The discrepancy between the energy input and rise of the mixture temperature

We identify two key processing steps in pellet manufacturing where significant heat is transferred into the particle mixture (see Fig. 1). The first heating step occurs during steam conditioning. During steam conditioning, heat exchange takes place between hot steam and cold particles. This process is driven by the temperature difference between the particles and the injected steam [13]. The specific energy transferred during this process (e_C in J kg^{-1}) is determined by the steam's enthalpy (H_{vap} in J kg^{-1}), the in-

jection rate of the steam (Q_s in kg s^{-1}) and the feeding rate of the cold particles into the conditioner (Q_p in kg s^{-1}).

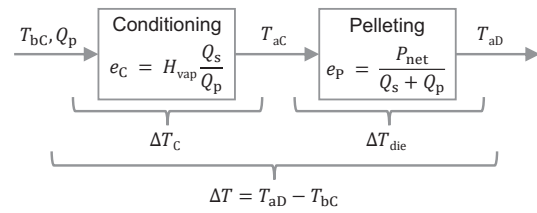


Figure 1. Schematic of the pelleting process and its heat transfer steps during steam conditioning and pellet extrusion. The temperature of the ingredients is increased in two consecutive steps, to reach process temperatures above T^* (see [8]).

The second heating step occurs during the pelleting process itself, where heat is dissipated through friction of the compressed extrudate within the extruder channel. This is due to inter-particle friction and particle-wall friction, as the particles are confined into the ring die's channels and from sliding friction as the pellets are extruded through the die channel. The specific energy transferred during this process (e_P in J kg^{-1}), is determined by the net power provided by the presses' engine (P_{net} in J s^{-1}) divided by the mass flow rate of the material, the combined weight of the condensed steam and the biomass mixture after conditioning ($Q_s + Q_p$ in kg s^{-1}).

The energy absorption during pelleting follows the principles of reactive extrusion. The rise in the product's temperature (ΔT) is proportional to the total specific energy transferred during the processing step (e) assuming that no energy is lost between the processing steps and can be estimated using Eq. 1:

$$e = e_C + e_P = c_p \Delta T + h \quad (1)$$

This equation accounts for the physical and chemical reactions that may occur in the grains and on the grain contacts during extrusion, as noted in [14], where e represents the total specific heat energy (J kg^{-1}) transferred during the process, c_p is the specific heat capacity of the ingredient mixture ($\text{J kg}^{-1} \text{K}^{-1}$), ΔT is the temperature change in the material during the heat transfer process, and h accounts for the contributions from endothermic ($h > 0$) and exothermic ($h < 0$) irreversible reactions (J kg^{-1}).

During steam conditioning, the specific energy required to increase the particulate material's temperature is proportional to its heat capacity and the temperature difference ($e_C = c_p \Delta T_C$, e.g., see Fig. B.1. in ref. [8]). The energy balance during steam conditioning can be calculated using Eq. 1, assuming $h = 0$ and c_p as constant. This is consistent with existing literature, which shows that the short retention time, low moisture content (10–16%), and atmospheric pressure are not likely to induce significant physicochemical changes in starchy ingredients [19–22]. To the best of our knowledge, no data is currently available on physicochemical transitions in other functional ingredient groups during steam conditioning.

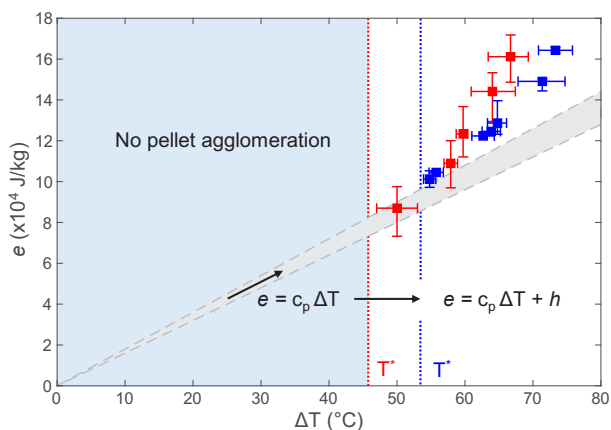


Figure 2. Above T^* , a surplus of energy (e) is required to further increase the material’s temperature, as part of the ingredients undergo phase transitions and start to agglomerate. Two independent pelleting trials were performed, pelleting a biomass mixture containing granular maize-sugar beet pulp 50/50% w/w. The steam conditioning temperature was varied systematically between approximately 34°C and 85°C. Red squares correspond to pellets produced with initial temperatures $T_{bc} \approx 24^\circ\text{C}$, and blue squares correspond to pellets produced with initial temperatures $T_{bc} \approx 12^\circ\text{C}$, resulting in two distinct T^* values. The values for e correspond to the sum of e_c and the net mechanical energy used by the press (see Methods). The values for e increase sharply as the process temperature rises above T^* , indicating a shift in the heat absorption mechanism. This behavior contrasts with processes where no physicochemical reactions occur, as shown by the gray region. The energy associated with pure heating of the material was calculated using Eq. 1, with c_p values ranging from 1.6 to 1.8 $\text{kJ kg}^{-1} \text{K}^{-1}$, consistent with literature values [15–18].

However, it is reasonable to assume that the process conditions—characterized by short retention times and low moisture levels—are similarly unfavorable for such transitions in these materials.

Steam conditioning is followed by the pelleting step in which the loose ingredient particles are transformed into mechanically rigid pellets. Looking at the overall process, we observe that the total specific energy required to heat the material (e) increases abruptly when the T_{ad} reaches values above T^* (see Fig. 2). This increase is attributed to physicochemical transitions that are known to occur during pelleting, such as protein denaturation and starch gelatinization [19, 20, 22–24]. These transitions require additional energy to drive changes in the material’s molecular structure, classified as either first-order phase transitions or second-order state transitions (for detailed descriptions see e.g., [25–28]).

In a first-order phase transition, such as melting, the enthalpy (h) exhibits a stepwise change at the transition temperature, representing the energy absorbed or released during structural transformations. Additionally, the heat capacity (c_p) may diverge at the transition temperature under ideal conditions. In contrast, during second-order phase transitions or a glass transition, the enthalpy changes continuously, while the heat capacity changes stepwise.

These effects can be analyzed during a modulated DSC (MDSC) experiment, which is capable of separating the reversible (c_p) and irreversible (h) processes by analyzing the change in temperature while the heat flow is modulated. However, multiple phase transitions may occur within the biomass mixture in an overlapping temperature regime, which complicates the analysis [28, 29]. Fig. 3 shows a schematic representation of a DSC experiment, from which Δc_p and h can be determined.

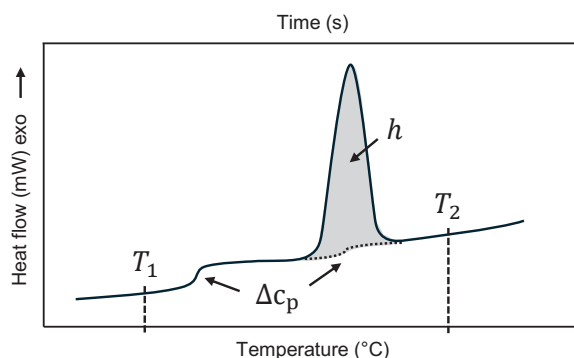


Figure 3. This schematic represents a result obtained during a DSC experiment. The changes in the heat flow at a given temperature and subject to an experimental (constant) heating rate (q), highlight changes in the physical and chemical structure of the material. This schematic indicates where Δc_p and h are determined, given that $c_p = \text{heat flow}/(q \times m)$, with m the mass of the sample. The two indicated temperatures, T_1 and T_2 , correspond to the hypothetical initial and final temperature of the material during pellet manufacturing.

In this work, we deviate from the approach proposed by Janssen *et al.* [14] by accounting for changes in c_p through the addition of an extra term: $\int \Delta c_p dT$. The effect of varying c_p on the specific energy absorption can be described by $\int c_p(T) dT$. However, experimental data indicates that c_p shows only marginal changes with temperature, except during phase transitions, where a measurable Δc_p is observed [30–32]. Consequently, in this manuscript, we presume c_p to be a constant unless phase transitions occur. The term $\int \Delta c_p dT$ represents the absorption of extra energy due to the effective change in c_p during a phase transition. The value for Δc_p then represents the average change in the heat capacity over the experimental temperature interval, represented by T_1 and T_2 in Fig. 3 (e.g., T_{bc} to T_{ac} for conditioning or T_{ac} to T_{ad} for pelleting) for which e is determined experimentally.

The effect of these physicochemical transitions on the absorbed energy during the pelleting process is shown in Fig. 2. The blue area represents the temperature regime where the process temperature remains below T^* , and pellet agglomeration does not occur. In this regime, the particles are purely heated while no physicochemical transitions occur, therefore the specific energy absorption is given by $e = c_p \Delta T$. Above T^* , the energy absorption mechanism changes, due to the occurrence of physicochemical transitions.

Near T^* , only small amounts of additional energy are required (data points remain close to the predicted heating values within the gray region), but pellets formed under these conditions exhibit low durability (low PDI). At higher temperatures, well-consolidated, high-PDI pellets are produced, accompanied by a progressive increase in the energy absorption (see Fig. 4).

We propose that above T^* , the specific heat absorption by the material increases as various physicochemical processes take place. These transitions, including protein denaturation and starch gelatinization, are believed to drive structural changes in the material, facilitating the agglomeration of individual particles and forming a well-consolidated pellet [28, 33].

In the next sections, we extend our pelleting framework to explain why agglomeration fails below T^* and why this temperature is the boundary for pellet formation. Our analysis focuses on the energy balance during extrusion, where phase transitions and bond formation occur. Specifically, we examine the conversion of mechanical energy supplied by the pellet press into heat and its subsequent impact on pellet durability.

3 Energy conversion during pellet agglomeration

During the pelleting step, a portion of the mechanical power supplied by the electrically driven motor(s) is converted into heat through friction. This heat is conducted into the ingredient mixture, raising its temperature by ΔT_{die} (see Fig. 1), while some heat is transferred to the metal components of the press, including the pelleting die. The heat absorbed by the pelleting press is subsequently dissipated to the environment via radiation and convection. Once the pelleting die reaches a steady-state temperature, the heat fluxes stabilize. This process is described by Eq. 2 modified from [14]:

$$P_{\text{zero}} + P_{\text{net}} = Q_v \left(\rho (c_p \Delta T_{\text{die}} + \int_{T_{ac}}^{T_{ad}} \Delta c_p dT) + \rho \Delta e_{\text{irr}} \right) + P_{\text{loss}} + P_{\text{zero}} \quad (2)$$

Here, P_{zero} represents the baseline power consumption of the motor(s) when running without any load from the material being processed—essentially the energy required to keep the equipment operating in an idle state (W). P_{net} is the net mechanical power converted to heat (W, see Fig. 1), Q_v represents the volumetric flow rate of the powder mixture through the die (m^3s^{-1}), ρ is the density of the agglomerated pellet within the die (kg m^{-3}), c_p stands for the baseline specific heat capacity of the ingredient mixture before phase transitions occur, Δc_p stands for the change in heat capacity during a phase transition averaged over the temperature interval T_{ac} to T_{ad} which is equal to ΔT_{die} , thus ΔT_{die} denotes the temperature change of the particle mixture over the extrusion die (K, see Fig. 1), Δe_{irr} represents the irreversible change in the materials energy content after a phase transition (e.g., after gelatinization of starch) (J kg^{-1}), and P_{loss} indicates heat losses to the

environment (e.g., through convection, conduction, or radiation) (W).

The sum on the left-hand side ($P_{\text{zero}} + P_{\text{net}}$) corresponds to the total power used by the press (P_{tot}), but P_{zero} does not contribute to heat production within the press and thus appears on the right-hand side of the equation as well. In case the heat losses (P_{loss}) are absent or at least negligible in comparison to the first term on the right-hand side, the adiabatic condition holds, and the temperature rise of the pellet during extrusion can be calculated using Eq. 3 (similar to equation 9 in ref [14]):

$$\text{SME}_{\text{net}} = \frac{P_{\text{net}}}{Q_v \rho} = c_p \Delta T_{\text{die}} + \int_{T_{ac}}^{T_{ad}} \Delta c_p dT + \Delta e_{\text{irr}} \quad (3)$$

Here, the left-hand term represents the net specific mechanical energy (SME_{net} in J kg^{-1}), which indicates the proportion of mechanical energy from the motor converted into heat per unit mass. On the right-hand side, we find terms corresponding to the heating of the ingredient mixture, the change in heat capacity during a phase transition averaged over the experimental temperature interval ΔT_{die} , and the irreversible energy absorbed by physicochemical transitions (Δe_{irr} , which Janssen *et al.* [14] refer to as h) such as protein denaturation and starch gelatinization [25]. During these transitions, energy is irreversibly absorbed as the molecular structure changes, acting as an effective heat sink in the pellet extrusion process.

Based on experimental pelleting data, we provide evidence that physicochemical transitions are critical for generating the stickiness needed to bind particle mixtures into durable agglomerates. We emphasize that such processes may only occur at the contacts of the particles.

In our analysis, we reformulate Eq. 3 by dividing all terms by ΔT_{die} :

$$\frac{\text{SME}_{\text{net}}}{\Delta T_{\text{die}}} = c_p + \langle \Delta c_p \rangle + \frac{\Delta e_{\text{irr}}}{\Delta T_{\text{die}}} \quad (4)$$

Here, $\langle \Delta c_p \rangle$ is the average change of the heat capacity over ΔT_{die} due to phase transitions. Equation 4 allows us to compare two scenarios:

1. Minimal physicochemical transitions ($\Delta e_{\text{irr}} \approx 0$ and $\Delta c_p \approx 0$): Here, the majority of the mechanical energy is used to increase the materials temperature according to $\text{SME}_{\text{net}} = c_p \Delta T$. In this case, little to no inter-particle bond formation occurs, resulting in a low PDI or the production of dust if $T_{\text{ad}} < T^*$ (see Benders *et al.*).

2. Significant physicochemical transitions ($\Delta e_{\text{irr}} > 0$ and $\Delta c_p \neq 0$): Here, physicochemical transitions increase energy absorption (e.g., starch gelatinization is an exothermic process [34]), inducing stickiness and enabling inter-particle bonds. This results in higher PDI values as $\text{SME}_{\text{net}}/\Delta T_{\text{die}}$ increases [9, 10].

Fig. 4 shows how the fines fraction—representing the fraction of pellets that do not remain intact during a durability test ($1 - \text{PDI}$)—decreases as the ratio of $\text{SME}_{\text{net}}/\Delta T_{\text{die}}$ increases.

The first case, in which only limited physicochemical transitions are expected, can be found on the left-hand side of Fig. 4. Under these process conditions, the T_{ad} of

the pellets is slightly above the T^* of the mixture. The $SME_{net}/\Delta T_{die}$ -ratio approximates the value of the c_p of the ingredient mixture before significant physicochemical transitions take place, here experimentally estimated between 1.4 and 1.6 $\text{kJ kg}^{-1}\text{K}^{-1}$. In this case, only a small amount of energy is absorbed by the reactions inside the material, resulting in minimal cohesiveness between the particles and a low PDI.

In contrast, high PDI pellets (≥ 0.95) are found on the right-hand side of Fig. 4. At higher T_{ad} and ΔT , the $SME_{net}/\Delta T_{die}$ -ratio rapidly increases due to contributions from Δc_p and Δe_{irr} , reflecting abundant transitions that create strong inter-particle bonds. We conclude that the occurrence of physicochemical transitions, reflected by the energy absorption by the material, are essential for the initial inter-particle bond formation in pellets.

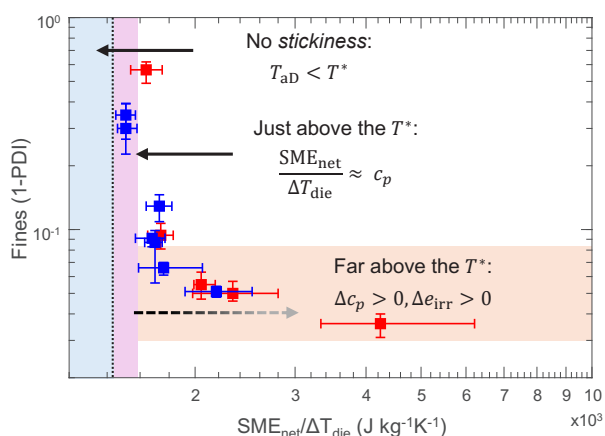


Figure 4. Physicochemical processes within the ingredients cause the absorption of additional energy during the pelleting process and are required to initiate the agglomeration process. Two independent pelleting trials were conducted, varying the steam conditioning temperature such that T_{ac} ranged between 34°C and 85°C, using a biomass mixture of maize and sugar beet pulp (50/50% w/w). The color coding for these trials corresponds to Fig. 2. The blue region denotes where pellet production fails due to insufficient agglomeration ($T_{ad} < T^*$). In the magenta region, just above the stickiness threshold T^* , only a limited amount of absorbed energy contributes to forming sticky bonds, with $SME_{net}/\Delta T_{die}$ approximating c_p . As T_{ad} increases beyond T^* (orange region), additional energy is absorbed through physicochemical processes, enabling inter-particle bond formation. This energy absorption correlates with a reduced fines fraction after durability tests and an increase in PDI. Note the logarithmic scales on both axes.

4 Consequences for particle-based reactive extrusion optimization

The results presented in Fig. 4 may have limited applicability when liquid binding agents like molasses are introduced during the pelleting process. Molasses, a highly viscous and sticky material, facilitates inter-particle bonding through mechanisms distinct from the temperature-dependent stickiness of ingredients defined by T^* [35–

37]. However, for mixtures lacking such viscous binding agents, heating alone during pelleting is insufficient to achieve significant particle cohesion. Therefore, precise process control is required to ensure that T_{ad} exceeds the value of T^* , enabling the formation of inter-particle bonds through physicochemical transitions, necessary for producing durable pellets.

However, aligning the reaction rates of physicochemical transitions with other process conditions—such as the die residence time and forces exerted on the material—is complex but necessary for inducing sufficient inter-particle bonding [9]. Achieving this alignment is far from trivial due to the complexity of the pelleting process.

The ingredient mixtures used inside a pellet mill are heterogeneous; therefore, multiple transitions can take place simultaneously as the particulate ingredients are pelleted. These transitions are time-dependent, often involving a spectrum of slow rate constants under low-moisture conditions [21, 38], and are influenced by process parameters such as temperature, local moisture content, and die residence time [19, 20, 39]. For instance, only approximately 20–50% of native starch gelatinizes during the process, depending on the process conditions and analysis methods [19, 20, 22, 23]. Note that the reactive extrusion step therefore also induces a change in the nutritional value of the ingredients in case the biomass is food or feed [24], which can seriously affect the dietary impact of the extrudate for humans and other (farm) animals. Water added during the process acts as a plasticizer, lowering the temperature required for these transitions [40]. However, the hydrothermal conditions inside the pellet press are highly non-uniform, with a significant proportion of water localized near the pellet surface [7]. Consequently, differences of up to 25% in the degree of gelatinization have been observed between the inner and outer regions of a single pellet [20].

From Fig. 4, we observe that the energy absorption increases by approximately $0.7 \text{ kJ kg}^{-1}\text{K}^{-1}$ as the ingredients transform from a loose powder (at approximately $1.5 \text{ kJ kg}^{-1}\text{K}^{-1}$) into well-agglomerated pellets (e.g., $PDI \geq 0.95$ at approximately $2.2 \text{ kJ kg}^{-1}\text{K}^{-1}$). This increase is attributed to two factors: the change in heat capacity (Δc_p) and the enthalpy of irreversible transitions ($\Delta e_{irr}/\Delta T_{die}$).

To estimate the contributions of Δc_p and Δe_{irr} to increase in $SME_{net}/\Delta T_{die}$, we rely on the limited available literature under relevant processing conditions. We focus on starch as the primary reactive component in the mixture because systematic data for other functional ingredient groups is unavailable. Starch typically exhibits Δc_p values due to the glass transition ranging from 0 to $0.4 \text{ kJ kg}^{-1}\text{K}^{-1}$ [30–32]. Assuming that approximately 50% of the starch undergoes phase transitions, the overall contribution of Δc_p is estimated to be around $0.2 \text{ kJ kg}^{-1}\text{K}^{-1}$.

The remaining $0.5 \text{ kJ kg}^{-1}\text{K}^{-1}$ corresponds to $\Delta e_{irr}/\Delta T_{die}$. Accounting for the temperature change across the die ($\Delta T_{die} \approx 15$ to 25°C), this value translates to an enthalpy range consistent with typical starch gelatinization values of 5 to 20 kJ kg^{-1} [14, 21, 27, 30, 32, 41, 42].

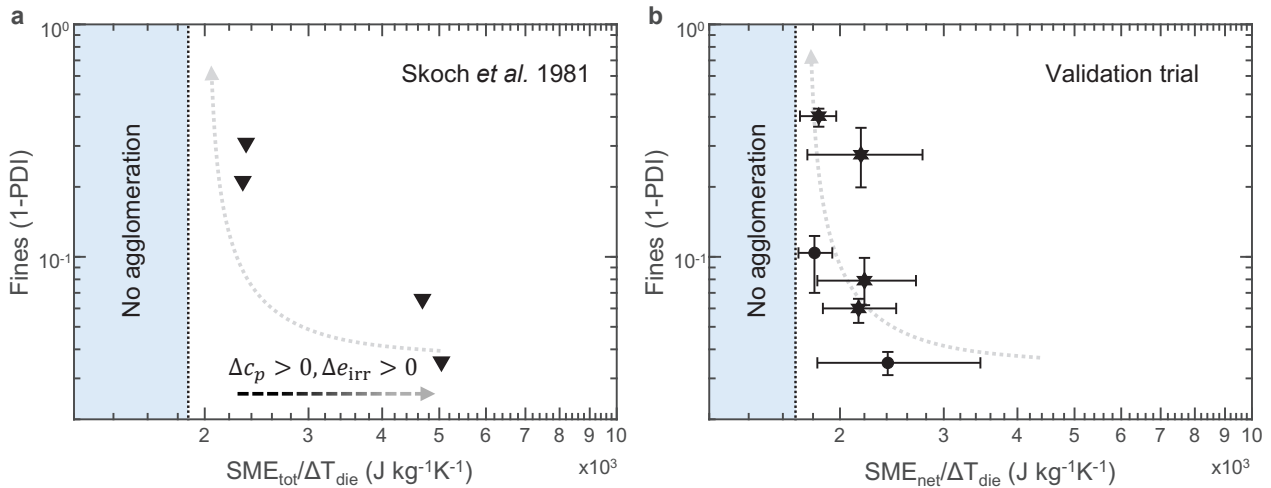


Figure 5. The absorption of mechanical energy by physicochemical transitions facilitates the agglomeration of ingredients into durable pellets. We validated our hypothesis by analyzing data from Skoch *et al.* [19] (a) and data from a validation trial using a mixture of maize, soy bean meal, and oat hulls (17, 33, and 50% w/w, respectively). This validation trial involved systematically varying the steam conditioning temperature (T_{ac}) and using two pelleting dies with 6 mm-diameter holes and L/D ratios of 6 (stars) or 9.3 (spheres) (b). In both cases (a) and (b), part of the mechanical energy was converted through physicochemical transitions during pelleting, indicated by the simultaneous increase in the $SME_{net}/\Delta T_{die}$ ratio and reduction of the fines fraction.

The heterogeneous chemical composition of feed mixtures, combined with non-uniform hydrothermal conditions during reactive extrusion of pellets, poses daily challenges for process operators. These complexities can be addressed in future studies focusing on the specific contributions of $e/\Delta T$ for the overall process or $SME_{net}/\Delta T_{die}$ during the mechanical processing of the ingredients. By using mixtures with well-characterized chemical compositions, the role of Δe_{irr} can be interpreted more accurately. Such studies will improve our understanding of the extent to which physicochemical transitions need to be induced to improve extrudate quality, e.g., while minimizing energy use and emission of carbon. In the following section, we reflect on the presented framework and demonstrate that the findings shown in Fig. 4 are not limited to the binary ingredient mixture studied here but are applicable to a broader range of biomass compositions.

5 Validation of the conceptual framework

The generality of our presented framework on the effect of physicochemical transitions on energy absorption during the pelleting process, in relation to the formation of durable pellets, is demonstrated by analysis of the experimental data obtained by Skoch *et al.* [19] (see Table 1), who pelletized an animal feed diet (for poultry), and by analyzing our own experimental pelleting trial using a different ingredient mixture.

The ingredients used in the biomass mixture directly determine the mixture’s physicochemical properties (e.g., see [23, 33, 43]). In [8], we have shown that the T^* of the Skoch mixture (see Table 1 in ref. [19]) and our validation mixture (composed of maize, soy bean meal and oat hulls of 17, 33 and 50% w/w respectively), varies from the reference maize-sugar beet pulp mixture (50/50% w/w)

due to the differences in chemical composition. Each particle mixture has its own unique agglomeration temperature (T^*) at which initial inter-particle bonds are formed. However, the underlying mechanism of particle bond formation should be consistent across mixtures, provided no viscous binders such as molasses are added. For particles to transition from non-sticky to sticky, biomass ingredients must undergo physicochemical transitions, which is reflected by the increase in the $SME/\Delta T_{die}$ -ratio while the fines fraction reduces (Fig. 5a&b).

In Fig. 5a, the gross (total) specific mechanical energy (SME_{tot}) was used to calculate $SME_{tot}/\Delta T_{die}$, rather than the net specific mechanical energy, since Skoch *et al.* [19] reported gross mechanical energy use ($P_{zero} + P_{net}$). Changes in the production rate would confound with the calculation, so we specifically selected the data corresponding to a production rate of 655 kg/h for our calculations (see Table 1). However, the calculated $SME_{tot}/\Delta T_{die}$ values are shifted to higher values due to the contribution of the idle load (P_{zero}), which remains constant and independent of the production rate.

Due to the limited dataset—only four runs were conducted at a constant production rate—the results presented in Fig. 4 and Fig. 5a can only be qualitatively compared. However, the observed increase in $SME_{tot}/\Delta T_{die}$, coupled with a simultaneous decrease in the small particle (fines) fraction, suggests that physicochemical changes play a key role in the formation of inter-particle bonds.

The data from the validation experiment is presented in Fig. 5b. During this experiment, a decrease in the fines fraction was observed as the energy absorbed by the material increased, suggesting the occurrence of physicochemical changes during the pelleting process. However, the relationship between $SME_{net}/\Delta T_{die}$ and the fines fraction appears weaker, indicating that additional factors may in-

Before conditioning (°C)	After conditioning (°C)	After pelleting (°C)	Production rate (kg/h)	Pellet durability (%)	SME _{tot} (kWh/t)
27	27	68	655	79.1	26.4
27	65	75	655	93.5	13.0
27	80	87	655	96.5	9.8
21	21	63	655	69.5	27.4
21	65	72	1636	90.6	10.9
21	78	83	1800	93.8	9.0

Table 1. Combined production data reported by Skoch *et al.*, see Tables II and IV in [19].

fluence agglomerate quality (e.g., aging [44]). Despite this, the lower limit for agglomeration in this biomass mixture—the asymptotic value of c_p —was estimated at approximately $1.7 \text{ kJ kg}^{-1}\text{K}^{-1}$. This value, along with the estimated c_p of approximately $1.5 \text{ kJ kg}^{-1}\text{K}^{-1}$ for the reference mixture, aligns well with literature values for c_p [15–18].

From these results we confirm that physicochemical transitions are essential in the transition from a loose particle mixture into a well-agglomerated extrudate. Insufficient heating of the material, below their T^* results in the production of non-agglomerated ingredients due to a lack of inter-particle cohesion. Therefore, T^* sets the lower temperature limit in the pelleting process. However, the temperature at which these transitions are initiated are subject to the ingredient composition. We believe that, by introducing this framework, a significant advancement of our understanding of the functional ingredient properties can be made. These advancements can be made by systematic monitoring of the $\text{SME}_{\text{net}}/\Delta T$ -ratio over a wider range of process data, and by evaluating the effect of T_{ad} in relation to how pellet durability changes.

6 Summary, considerations and outlook

In this study, we expanded on the framework established in our previous work, which identified T^* as the lower temperature limit for pellet agglomeration. This work provides new insights into why pellet agglomeration occurs when process temperatures exceed T^* by analyzing the $\text{SME}_{\text{net}}/\Delta T$ ratio. This ratio quantifies the energy absorbed by the ingredients as their temperature increases during the process. We have improved our understanding of the underlying mechanism that initiates inter-particle bond formation during pellet manufacturing. Specifically, we now recognize T^* as the temperature at which physicochemical transitions begin, coinciding with a simultaneous increase in $\text{SME}_{\text{net}}/\Delta T$ and pellet durability. Thus, we classify pellet manufacturing as a reactive extrusion process, where physical and chemical changes in the involved particles are essential for the particle agglomeration process.

Our findings demonstrate that, below T^* , c_p governs the predictable increase in material temperature during standard heating conditions, such as steam conditioning,

during which a non-significant amount of physicochemical changes is observed [19, 20, 22]. However, during pellet extrusion various physicochemical processes take place such as starch gelatinization or protein denaturation, which alter the energy absorption significantly through the contributions of Δe_{irr} and Δc_p . This rapid increase of the energy absorption above T^* together with the simultaneous increase in PDI, implies that T^* is indicative for the temperature at which the materials transition from a non-sticky into a sticky state [9, 10]. These insights result in a more precise understanding of how tuning the temperatures within the pelleting process impacts physical pellet quality.

However, several important questions remain unanswered. One key unknown is the specific number and types of bond-forming reactions required to produce high-quality pellets. Our results suggest that PDI improves with increasing energy investment. However, we envision that for the particles to stick together, the transitions should occur at the inter-particle contact sites, but not necessarily within the particles themselves, hence only a small amount of energy may be required to generate the required initial stickiness. Another key aspect is the effect of the ingredient composition at various processing conditions, while different ingredients have different functional properties which may or may not show similar behavior during and after the pelleting process. However, we have now provided a new set of tools to analyze the behavior in order to understand such ingredient specific characteristics. Future work could systematically explore how properties such as moisture content, protein content, co-product inclusion, and starch type affect the physicochemical properties of the ingredient mixture and how these variations influence the T^* . Moreover, the effect of die residence time relative to the rate at which reactions occur may play a critical role in optimizing mill performance [39]. Ultimately, this will enable us to balance the heat transfer processes accordingly. Certain products may benefit more from steam-induced heating, while others require the heat generated through friction during extrusion to produce a durable pellet.

Our study paves the way for a more ingredient-specific approach to optimizing particle-based material extrusion processes. The physicochemical changes induced during pelleting can alter the nutritional value of feed [24, 45],

establishing a direct connection between excess absorbed energy ($SME_{\text{net}}/\Delta T_{\text{die}}$) and nutritional changes. Future research could further validate the effectiveness of our analysis method in predicting and quantifying these changes. Additionally, the relationship between the energy absorption and T^* provides a powerful tool for tailoring processes to the raw materials, as well as for adjusting conditions with additives that promote agglomeration by lowering T^* . Understanding and controlling these ingredient-specific physicochemical reactions could pave the way for more efficient and adaptable pelleting processes. We envision that by doing so, we could enhance the energy efficiency and better control product quality across a wide variety of products ranging from animal feed to bioplastics and municipal waste. By incorporating the reactive contributions, we have laid the foundations for both future scientific exploration and industrial innovation, especially as the field evolves toward more complex particle-based ingredient mixtures.

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A Methods

A.1 Pelleting trials

Pellets were produced using an RMP200 ring-die pelletizer (Münch Edelstahl GmbH, Hilden, Germany). Steam conditioning was performed as described in Benders *et al.* [7] using high-quality dry steam to heat the cold ingredient mixture (T_{bc}) to a specified T_{ac} . The pelletizer was configured with two rollers and equipped with ring dies featuring 6-mm diameter holes and compression ratios (L/D) of 6, 9.33, or 12, as detailed in Benders *et al.* [7].

Each trial began with a 20-minute steady-state period, after which approximately 2 kg of pellets were collected, cooled using ambient air for 10 minutes, and stored in plastic bags at 4 °C until analysis. Sampling was performed 2–3 times per trial at 15–20-minute intervals.

Process parameters, including T_{bc} , T_{ac} , T_{ad} , and moisture content (mc) before conditioning, after conditioning, and post-extrusion, were recorded. Electrical current and production rate (kg/h) were also monitored. From these, the change in moisture content (Δmc , in kg water per kg ingredients), the temperature increase during conditioning (ΔT_{C}), and the temperature change over the ring die (ΔT_{die}) were calculated (see ref. [7]).

The specific energy transferred during conditioning (e_{C}) was calculated using Δmc and the latent heat of vaporization of water (2.26 MJ/kg, assuming 100% efficiency). Specific mechanical energy ($SME = e_{\text{P}}$), including SME_{tot} and SME_{net} , was determined as per Bastiaansen *et al.* [46], where SME_{net} accounts for idle-load correction. Average e values presented in Fig. 2 are sums of mean e_{C} and SME_{net} , while the range reflects combinations of minimum and maximum values for e_{C} and SME_{net} .

Error bars for ΔT in Fig. 2 represent minimum and maximum values for $T_{\text{ad}} - T_{\text{bc}}$, while those for ΔT_{die} represent $T_{\text{ad}} - T_{\text{ac}}$. For $SME/\Delta T_{\text{die}}$, minimum and maximum values were calculated by dividing SME and ΔT_{die} extremes, respectively, where the minimum is given by dividing the minimum value for SME by the maximum value for ΔT_{die} and vice-versa.

A.2 Materials

A.2.1 Maize-sugar beet pulp reference mixture

A 50/50% w/w mixture of corn kernels and low-sugar sugar beet pulp was purchased from Research Diet Services (Wijk bij Duurstede, The Netherlands) as specified in Benders *et al.* [7]. Trials were conducted during summer ($T_{\text{bc}} = 24.3$ °C) and winter ($T_{\text{bc}} = 11.8$ °C), with differences attributed to ambient air temperature. Pelleting was performed at approximately 250 kg h⁻¹ using a ring-die with $D = 6$ mm and $L/D = 12$, and steam conditions as mentioned in the caption of figures 2 and 4.

A.2.2 Validation trial mixture

The validation mixture contained maize, soy bean meal (high-protein) and oat hulls (17, 33 and 50% w/w respectively), was ground over a 4-mm hammer mill screen and purchased from Research Diet Services. Pelleting was conducted at a production rate corresponding to a die residence time of approximately 9 s with the die specifications given in Fig. 5, corresponding to 260 kg h⁻¹ for the $L/D = 9.3$ and 170 kg h⁻¹ for the $L/D = 6$. The ΔT_{C} was varied between 45 °C to 75 °C.

A.3 Physical pellet quality

Pellet durability was analyzed using a Holmen NHP100 tester (Tekpro, USA) with a 3-mm screen, following Benders *et al.* [7]. The pellet durability index (PDI) was converted into fines fraction ($1 - \text{PDI}$). Error bars for fines fraction represent the range of experimental PDI values.

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