

Effect of screw speed and plasticizer on the torque requirement in single screw extrusion of starch based plastics and their mechanical properties

Madhusweta Das

Department of Agricultural and Food Engineering, Indian Institute of Technology, Kharagpur 721 302, India
Email: madhu@agfe.iitkgp.ernet.in

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Rod-shaped plastics have been prepared from blends of corn starch (69%) and plasticisers (31%), in the form of glycerol and water in different proportions (ranging from no glycerol to full glycerol) using a single screw extruder (compression ratio 1:1) (Length: Diameter ratio of 20:1) operated at specified constant temperatures, viz., 70, 110 and 110°C at feed, mixing (plasticizing) and metering zones of the barrel, respectively. The screw speed was varied between 20 and 80 rpm. The torque generated during extrusion was recorded and then ultimate tensile strength (UTS) and elongation at break (EB) were measured after storing the extrudates for nine months at ambient condition. There was no significant difference in torque requirement for extrusion for use of zero to 9% glycerol content in the blends; however, for glycerol content greater than 9%, the developed torque gradually increased over the full range of rpm used. The range of torque was from 4.5 to 28 Nm, and for each blend it passed through a minimum at 60 rpm. Ultimate tensile strength followed a decreasing trend with increase in glycerol content in the plasticizer mix. However, elongation at break followed an increasing trend for increase in glycerol content up to 24%, and thereafter followed a decreasing trend with further increase in glycerol content. For glycerol content less than 24%, screw speed had a strong and direct bearing on UTS, but for EB, screw speed had no specific trend.

Keywords: Single screw extrusion, Plasticizer, Starch based plastics

Use and throw non-biodegradable food packages made from petroleum based synthetic plastics are a matter of concern from ecological view point. To develop biodegradable alternative, extrusion processing, mainly twin screw extrusion of native starch in presence of plasticizers in the form of glycerol and water for production of thermoplastic starch has been the subject of intensive research over the last decade or so¹⁻³. It has been reported that thermoplastic starch is a complex system from the point of view of gelatinization, depolymerization and its interaction with the plasticizer, whereby the hydrogen bonds in native starch molecules disappear and new hydrogen bonds develop between the polar hydroxyl groups of water (and/or glycerol) and glucose units in the polymer chain⁴⁻⁷. The whole process is controlled by the shear action and temperature in the extruder, as well as on the feed composition⁸⁻¹⁰. Though single screw extruders have economical advantage, simplicity of operation and trouble free maintenance^{11,12}, and are conventionally used for making texturised and cooked foods, as well as in the compounding of synthetic thermoplastics,

reports on fabrication of biodegradable plastics using single screw extruders are limited.

Due to hydrophilic nature, the mechanical properties of thermoplastic starch using water and glycerol as plasticizer are expected to be sensitive to changes in ambient humidity¹³. Therefore, in most of the studies⁸⁻¹⁰, the extrudates were allowed a specified storage period at constant temperature and humidity, thus providing the scope for equilibration and formation of reoriented starch chain molecules, possibly leading to crystallization and chain-chain association; the product qualities were then evaluated.

The present study was undertaken to investigate the effect of mixed plasticizer composition and screw speed on (a) the torque requirement during single screw extrusion of blends comprising corn starch as the base polymer, and a mixture of glycerol and water as plasticizers, and (b) the tensile stress and elongation of the extrudates after storing the same for nine months at ambient condition.

Experimental Procedure

Sixty nine per cent corn starch (commercial grade, purchased from local market) and 31% of a mixed

plasticizer, consisting of variable proportions (Table 1) of glycerol (Laboratory grade, Ranbaxy) and water (glass distilled) were blended in a domestic mixer/blender (Sumeet, Model SP16, India) for 5 min. The blend was then fed (flood feeding mode) in a single screw extruder (Brabender, Model DO-Corder E DCE 330, Germany) of length:diameter ratio of 20:1 and extruded under a compression ratio of 1:1. Blend of each composition, was extruded at four different screw speeds, viz., 20, 40, 60 and 80 rpm, respectively (Table 1). For each of the screw speed, discrete extrusion was run and in each run, steady state was allowed to reach before collecting samples. Typically, steady state was reached in 5-10 min. Temperatures of the extruder barrel were set at 70, 110 and 110°C (as referred by the manufacturer's catalogue) for the feed, mixing and metering zones, respectively. The value of torque in each case was noted. Rod-shaped extrudate through round-hole die (3 mm diameter) was collected and kept at 40°C for overnight in an incubator to remove any free moisture. The product (about a kg) was stored in open mouth polythene sachet at ambient condition for 9 months. The stored product was equilibrated at 50% relative humidity for 48 h before ultimate tensile strength (UTS) and per cent elongation at break (EB) (flexibility) were measured using Instron Universal Testing Machine (Instron, Model 1011, UK). Each extrusion was replicated thrice. From each lot of sample, five replications were taken for measurement of mechanical properties. Details of these methodologies have been described elsewhere¹⁴.

The mean values i.e., average of three replicated run for torque, and average of fifteen readings for both UTS and EB, along with corresponding standard deviations have been estimated. The effect of composition and screw speed on the respective mean value has been analyzed by analysis of variance (also called F-test) at 1 or 5% level of significance following the method used for a single factor

experiment (here either plasticizer or screw rpm varies, while all other experimental conditions remain constant) with completely randomized design (as all the observations were independent) with equal replications¹⁵. Significant F test helps to indicate that the observed difference among treatment means is real and not due to chance. With significant F test in the analysis of variance, least significant differences was evaluated at 1 or 5% probability levels which helps to decide whether a particular pair (or pairs) of treatment is significantly different or not¹⁵. All the statistical calculations were done using Microsoft Excel 2000.

Results and Discussion

From the extruder die land, the hot translucent starch melt came out in the form of soft flexible rod without any visible sign of puffing, as shown in Fig. 1. All the compositions produced extrudates of similar look. The translucent appearance and absence of puffing indicated that the polymer molecules in the extruded mass were gelatinized and perfectly aligned; probably the shear developed in the barrel and the die land helped in such alignment¹⁶. Flexibility of the stored extrudates were, however, relatively lower compared to that of the fresh one coming from the die land and found to be dependent on plasticizer composition.

Effect of plasticizer on torque development

Figure 2 shows the effect of plasticizer on the torque developed during extrusion at different screw speeds (F-test significant at 1% level). As it was a

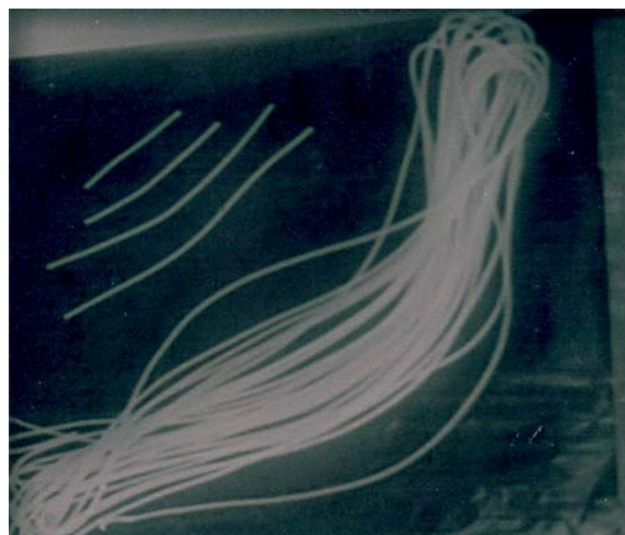


Fig. 1— Extrudate of starch-glycerol-water blend using single screw extruder

Table 1— Composition of blends extruded at various screw speeds

Blend No.	Composition, weight%		
	Starch	Glycerol	Water
1	69	0	31
2	69	9	22
3	69	18	13
4	69	24	7
5	69	31	0

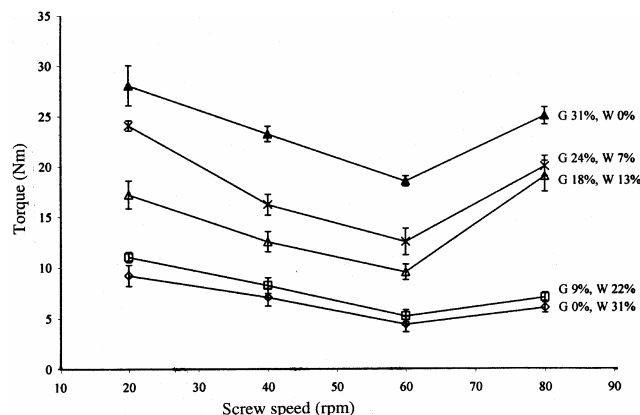


Fig. 2—Torque developed during extrusion of blends containing starch (69%), and varied amount of glycerol (G) and water (W) using different screw speeds

case of single screw extrusion and the material was introduced in flood feeding manner, it was quite likely that the mass inside the barrel propelled like a molten mass filling its entire annular space^{4,17}. Under this condition, at any screw speed the developed torque should depend on the viscosity of the melt^{4,18,19}. It is apparent that, the magnitude of torque corresponding to all the four screw speeds is lowest for the feed containing only water as the plasticizing agent. This might possibly be due to easy plasticization of the mass with this low molecular weight and less viscous plasticizer without exerting much drag and resistance to flow. With incorporation of 9% glycerol in the blend, torque showed insignificant [p (probability of failure) > 0.05] increase for all the rpm. With further inclusion of glycerol up to 31%, torque at any screw speed steadily increased ($p < 0.01$). However, at 80 rpm there was no significant increase with 24% glycerol from that of with 18% glycerol. The number of hydroxyl (-OH) groups available per unit weight of glycerol is less than that of water; incorporation of glycerol replacing equal weight of water, therefore, effectively reduces the number of -OH groups available for binding to starch⁷. Probably it decreased the extent of plasticization of the mass in the barrel that required higher value of torque⁴. It is worth mentioning that the torque required for processing of all the starch-glycerol-water mixture, in the present case, lies within 28 Nm which is comparable to the values reported earlier for extrusion of other starchy material using either single or twin screw extruder in food processing¹⁹⁻²¹.

Effect of screw speed on torque development

The torque required in extrusion usually should increase with increase in both, the viscosity of the

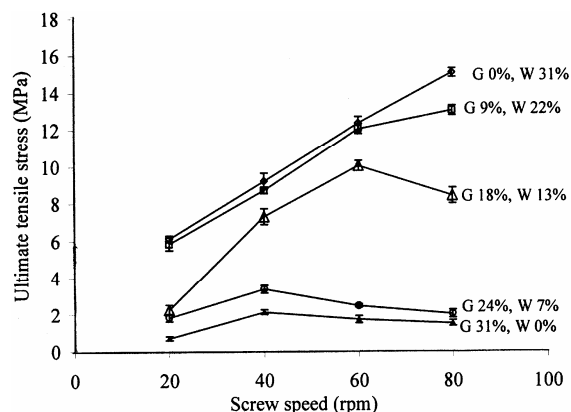


Fig. 3—Ultimate tensile stress of the extrudates prepared from blends containing starch (69%), and varied amount of glycerol (G) and water (W) using different screw speeds

melt and the screw speed^{4,18,19}. Here, for each composition variation of screw speed significantly changed the torque; however, some contradictory behaviour was observed. It gradually decreased with increase in screw speed from 20 rpm and attained the minimum value at 60 rpm (Fig. 2). As with full barrel, shear rate gained by the material was supposed to become proportional to the screw speed, possibly shear thinning effect and increasing plasticization for higher mixing lowered the viscosity of the mass^{19,22,23} which overruled the effect of increasing rpm^{20,24} for increasing the torque. When the screw speed was increased beyond 60 rpm, probably there was no further reduction in viscosity and the torque increased in the usual way^{4,24}.

Effect of plasticizer on UTS of extrudate

Figure 3 shows the effect of plasticizer (F-test significant at 1% level) on UTS of the extrudates. UTS was highest of the products prepared with water as the only plasticizer for all the screw speeds. For each increment of glycerol addition to make it up to 31% in the blend, UTS decreased significantly ($p < 0.01$); however, a few exceptions were noted *viz.*, when extruded at 60 rpm there were no significant differences for the products from the blend between 0 and 9% glycerol content, and between 24 and 31% glycerol content. Both water and glycerol plasticize the starch, but water being volatile may escape during storage, whereas glycerol is non-volatile, and its incorporation leads to stable plasticization¹⁴. Plasticization decreases the cohesive force and increases chain pliability, and probably, these effects lead the extrudates for becoming weak in mechanical strength with the increasing amount of glycerol in the blends.

Effect of screw speed on UTS of extrudate

Screw speed significantly controlled (1% level) the UTS of the products made from each composition; the difference in UTS for any pair of screw speed was significantly different. It is observed (Fig. 3) that, the UTS of the products made with no and 9% glycerol increased almost linearly with the increase in screw speeds from 20 to 80 rpm; however, the proportional increase for screw speed between 60 and 80 rpm was less for the latter. With 18% glycerol content, UTS increased for up to 60 rpm followed by a drooping trend. For products made with 24% and 31% glycerol, the UTS attained the peak values of 3.3 and 2.1 MPa, respectively, at 40 rpm after which it decreased. From the nature of the figure it is evident that the effect of screw speed on UTS for products from blend with 24 and 31% glycerol was much less than that of the other three compositions. The increasing trend of UTS with rpm, for all the products, was possibly due to increasing shear action that helped in molecular alignment^{24,25}. On the other hand, the decrease in UTS beyond certain screw speed might be attributed to major fragmentation of starch with excessive working (mixing). Apparently, more was the glycerol in the blend, less was the screw speed required for obtaining the corresponding maximum UTS of the product, i.e., the starch might have been easily fragmented under action of low shear²⁶.

It may be worth mentioning that the UTS of the products made in the present case from the blend containing 69% starch, 18% glycerol and 13% water are comparable to that of starch based plastics produced by Forssell *et al.*¹³ using twin extruder and stored for 8 months (20°C and 50% RH), and by Krogars *et al.*²⁷ using high pressure reactor and stored for 9 months (25°C and 60% RH).

Effect of plasticizer on EB of extrudate

It is observed (Fig. 4) that for gradual increase of glycerol up to 24%, EB of the products at any screw speed increased significantly ($p < 0.01$) [at 20 rpm, EB for the products for 0 and 9% glycerol was not significantly ($p > 0.05$) different]. However, when the products were made with 31% glycerol, EB decreased. It was even lower compared to that of the products from 18% glycerol. Possibly the gradual increase in plasticization with increasing amount of glycerol was responsible for increasing elongation. However, the reason for low elongation for products from 31% glycerol as the only plasticizer was not apparent within the present experimental limits.

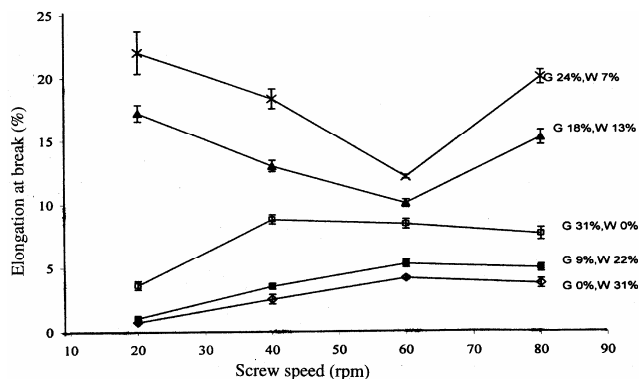


Fig. 4—Elongation at break of the extrudates prepared from blends containing starch (69%), and varied amount of glycerol (G) and water (W) using different screw speeds

Interestingly, this type of unexpected trend for elongation for the variation of plasticizer has earlier been reported by other workers²⁷⁻³⁰. It may be pertinent to mention that in amylose-glycerol-water system, a depressive effect on glass transition temperature was observed using glycerol quantities from 0 to 25%, but with more than 25% glycerol discontinuities were detected²⁸.

Effect of screw speed on EB of extrudate

For each blend, there was significant ($p < 0.01$ or $p < 0.05$) difference among elongation of the products obtained at different speeds; also the trend was different depending on the composition (Fig. 4). For 0 and 9% glycerol in the blends, EB increased for up to 60 rpm whereas for 31% glycerol it increased up to 40 rpm screw speed. In all these three cases, the increasing trend was followed by decreasing trend. The trends were opposite for blends containing 18 and 24% glycerol, i.e., EB decreased with increase in screw speed up to 60 rpm followed by an increasing trend. The present EB values of the products with 18% glycerol and 13% water are comparable to the values of starch based plastics reported by Krogars *et al.*²⁷.

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