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20 Mixing Index of a Starch Composite Extruder for Food Packaging Application

Adeshina Fadeyibi, Zinash Delebo Osunde, Gbabo Agidi and Egwim Chidi Evans

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Abstract: This encapsulates the performance of a single-screw extruder for use in the mixing and homogenization of the composite of cassava starch (1000 g), glycerol (45%–55% w/v), and zinc oxide nanoparticles (0%–2% w/v), based on the mixing index and rate constant. The machine was designed such that it can accommodate two diameters (5 and 7 mm) in the breaker plate; the output/1000 g of the starch composite was determined at a resident time of 5 min over a period of 1 h machine operation. The research revealed that the composite containing 55% w/v glycerol, 1% w/v zinc nanoparticles and 5 mm diameter of the holes in the breaker plate has the highest mixing rate constant ($k = 0.527$). Decay trends were observed as the mixing index increases with an increase in the resident time in the range of 45%–55% w/v glycerol concentration and a decrease in the output per 1000 g of the starch composite through both the 5 and 7 mm diameters of the hole of the breaker plate. The optimum output of the machine, whose desirability function is closer to the optimization goal, gave 0.53/1000 g. The amount of unmixed composite was significantly lower than the machine output and thus the single-screw extruder can be used to quantify and monitor mixing behavior of starch composites in the food industry.

20.1 INTRODUCTION: BACKGROUND

In industrial food process engineering, mixing is a unit operation that involves manipulation of a heterogeneous physical system with the intent of making it more homogeneous. Mixing occurs when materials are agitated inside the vessel (Hu and Chen, 2006). The nonuniformity of systems can be explained as a gradient of properties such as concentration, viscosity, temperature, color, concentration, phase, and temperature (Lee and Kwon, 1999). Mixing is due to the dispersion of components. It occurs in innumerable instances in the food industry and is probably the most commonly encountered of all process operations. There are, however, some aspects of mixing that can be measured and be of help in the planning and designing of mixing operations. Ideally, a mixing process begins with the components, grouped together in some container, but still separate as pure components. Thus, if small samples are taken throughout the container, almost all samples will consist of one pure component. The frequency of occurrence of the components is proportional to the fractions of these components in the whole container. As mixing proceeds, samples increasingly contain more of the components, in proportions approximating to the overall proportions of the components in the whole container. Complete mixing can then be defined as the state in which all samples are found to contain the components in

the same proportions as were in the whole mixture. Actually, this state of affairs would only be attained by some ordered grouping of the components and be a most improbable result from any practical mixing process. The assessment of mixed small volumes, which can be taken or sampled, is what mixing measurement is all about. Mixing measurement, however, must reflect the way the sample compositions move from the initial state to the mixed state, and if the sample is so large that it includes the whole mixture, then the sample composition is at once the mean composition and there remains no mixing to be done. At the other end of the scale, if it were possible to take samples at the molecular size, then every sample would contain only one or other of the components in the pure state and no amount of mixing would make any difference. Between these lie all of the practical sample sizes, but the important point is that the results will depend on the sample size.

Convective and intensive mixing are two common processes occurring in powders and composites. In the case of convective mixing, materials in the mixer are transported from one location to another. This type of mixing process often leads to a less ordered state inside the mixer, and the unmixed components are evenly distributed through the matrix. Usually this type of mixing is applied for free-flowing and coarse materials, such as the starch composite in this investigation. In the convective mixing, however, there are several processes available from silo mixers to horizontal and conical mixers. When materials are cohesive, which is the case with fine particles and also with wet material, convective mixing is no longer sufficient to obtain a randomly ordered mixture. The relative strong interparticle forces, which are not broken up by the mild transportation forces in the convective mixer will help to form lumps. Impact or shear forces are necessary to decrease the lump size. Additionally, the mixing of particles varying substantially in size or in density presents special problems, as there will be gravitational forces acting in the mixer that will tend to segregate the particles into size and density ranges. In such a case, initial mixing in a mixer may then be followed by a measure of the un-mixing and so the time of mixing may be quite critical. Mixing is simplest when the quantities that are to be mixed are roughly in the same proportions. In cases where very small quantities of one component have to be blended uniformly into much larger quantities of other components, the mixing is best split into stages, keeping the proportions not too far different in each stage. In planning the mixing process, it would be useful to take analyses through each stage of mixing, but once mixing times had been established, analysis on the final product becomes necessary (Hu and Chen, 2006; Lindt and Ghosh, 1992).

A number of experimental researches and modeling of the mixing behavior of various types of extruders have been conducted (Khalifeh and Clermont, 2005; Zhu et al., 2007; Jiang and Zhu, 2008; Zhu et al., 2009). Hu and Chen (2006) established a three-dimensional isothermal flow model of kneading-disc elements for the tri-screw extruder with finite element method to emphasize particularly on the analysis of flow rules. On this basis, they further studied the mean mixing efficiencies of the tri-screw and twin-screw extruders using computational fluid dynamic package. The chaotic mixing mechanism in the

twin-screw extruder has been studied, but a very low performance, usually because of its relatively complex geometry and long development time, has been reported (Hu and Chen, 2006; Potente et al., 1994). Hence, the study of the performance of the single-screw extruder, with simpler screw geometry and shorter development time is expedient. Also, the previous studies hardly figured out the performance of the single-screw extruder in terms of the mixing rate and index. On the other hand, no information was available about the analysis, optimization, and modeling of the mixing rate and index of the output of the starch composite. Consequently, there is a need to understand the behavior of the single-screw extruder by optimizing the output and evaluating its mixing performance or efficiency with respect to the mixing rate and index. Furthermore, the studies on examining the effect of the diameter of the holes in the breaker plate on the mixing efficiency of the single-screw extruder are lacking. In this investigation, the Box–Behnken optimization tool of the response surface methodology (RSM) was employed to study the output of the starch composites from the extruder, thus ascertaining the influence of the diameter of the holes in the breaker plate on the extruder output. The objectives of the present communication are to evaluate the mixing index and rate of a single-screw extruder and to understand the special contributions of the diameter of the holes in the breaker plate and concentration of the glycerol and the nanoparticles on the output of the starch composite from the single-screw extruder for application in the food packaging industry.

20.2 MATERIALS AND METHOD

20.2.1 SAMPLE PREPARATION

20.2.1.1 Preparation of Cassava Starch

In this study, starch was prepared from 250 kg of a freshly harvested cassava root. The crop was peeled and soaked in a clean bowl containing water for 24 h after which it was ground into pastes. The ground pastes were then sieved using a muslin cloth and the resulting filtrates was left undisturbed for 24 h to allow the starch to settle at the bottom of the bowls. The prepared starch of the cassava was dried under the sun for 1 week and a moisture meter was used to monitor its moisture contents up to 2% (wb).

20.2.1.2 Preparation and Functions of Zinc Oxide Nanoparticles

The sol–gel method described by Surya (2012) was used to prepare the zinc oxide nanoparticle at the Crop Processing and Storage Laboratory of the Department of Agricultural and Bioresources Engineering, Federal university of Technology, Minna, Nigeria. This method involves the mixture of two homogeneous solutions. The first solution was prepared by adding 30 mL of distil water to 20 mL of triethanolamine with constant stirring while adding 2 mL (100 drops) of ethanol in drop wise. A 5.39 g of zinc acetate di-hydrate was added to 50 mL of distil water with continuous stirring, to obtain a 0.5 M solution, as the second homogeneous solution. The two solutions were mixed together in 500 mL beaker and

a solution ammonium hydroxide and 10 mL distil water was added, in drop wise, with continuous heating and stirring for 20 min. A white bulky solution was formed when the resulting solution is left for 30 min. It was then washed 8–10 times with distil water and filtered in a filter paper. The resulting residue was dried in the oven at 95°C for 8 h.

The nanoparticles are particles that have at least one dimension within the nanometer range (1–100 nm). The particles have antimicrobial efficacy, and so can help to prevent the growth of deteriorating microorganisms in the composites. Thus, when incorporated in the starch composite for use in the production of films in the food packaging industry, the nanoparticles help in enhancing the shelf lives of the packaged fruits and vegetables (Sorrentino et al., 2007). Nanoparticles are also capable of increasing mechanical properties, such as percentage elongation, tensile strength, and elastic modulus, of the resulting film. The enhancement of many properties resides in the fundamental length scales dominating the morphology and properties of these materials. The inorganic particles have at least one dimension in the nanometer (from 1 to 100 nm) range (Lagaron and Sanchez-Garsia, 2008). It means that a uniform dispersion of these particles can lead to ultra-large interfacial area between the constituents. The very large organic/inorganic interface alters the molecular mobility, the relaxation behavior, and the consequent thermal (temperature stability), mechanical, and barrier properties (water and gas permeability) of the resulting nanocomposite materials (Azeredo, 2009).

20.2.1.3 Preparation of the Starch Composite

The starch composite was prepared by adding analytical grade glycerol (45%–55%, w/v) and zinc oxide nanoparticles (0%–2%, w/v) to 1000 g of the prepared cassava starch. The mixtures were put together and fed into the hopper of the screw extruder, developed (Figure 20.1) for the mixing and homogenization of starch composites (Fadeyibi et al., 2014). The extent of the mixing operation of the extruder was investigated for 1 h at an interval of 5 min by weighing the output/1000 g at two separate diameters of the holes in the breaker plate (5 and 7 mm). The mixing indices and rates were computed to ascertain the performance in terms mixing efficiency of the screw extruder.

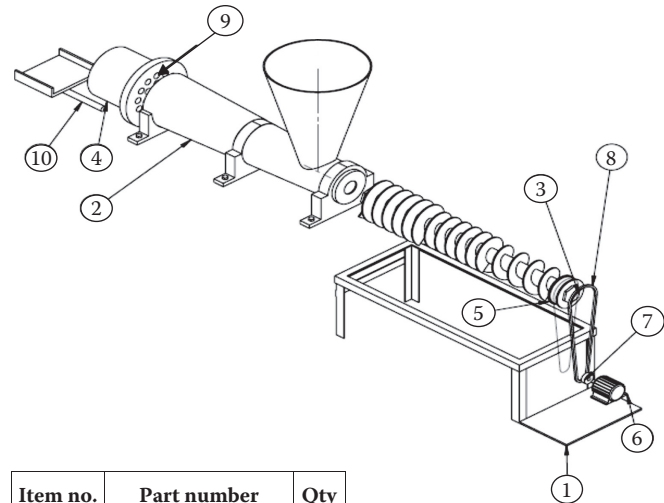
20.2.1.4 Mass of Starch Composite before Mixing

The required mass of the composite of cassava starch and glycerol to be mixed is essential in the determination of the mixing index of the machine. Assuming that the capacity of the extrusion conveyor is 15 tons/h and the expected time of operation is 30 min, which includes the time for feeding and discharging, the mass of the material mixed per ton was computed from Equation 20.1.

$$W_t = \gamma_c \left(\frac{V_c \times T_c}{T_e} \right) \quad (20.1)$$

where:

W_t required weight of composite (kg) of cassava starch and glycerol to be mixed



Item no.	Part number	Qty
1	Extruder seat	1
2	Extruder barrel	1
3	Screw/auger	1
4	Die	1
5	Pulley	1
6	Motor	1
7	Motor pulley	1
8	Belt	1
9	Ø of hole of breaker plate	1
10	Passage/collector	1

FIGURE 20.1 3D view of the starch composite extruder.

γ_c is the bulk density of the composite (kg/m³)

V_c is the volume of composite (m³) to be mixed

T_c is the time required to mix the composite fed in the metering zone of the extruder = 1 h (60 min)

T_e expected time to mix 1 tone of the composite = 5 min

20.2.2 DETERMINATION OF EXTENT OF MIXING OF INDIVIDUAL PARTICLES OF THE COMPOSITE

The level of mixing of individual particles for making the composites, namely the starch, glycerol, and zinc nanoparticles, was considered as a criterion for an effective mixing according to the mathematical relationship presented in Equation 20.2. It was assumed that all the particles of the composite were equally sized and that each particle was pure starch, glycerol, or zinc nanoparticles.

$$S^2 = \sum \frac{(x - \bar{x})^2}{n} \quad (20.2)$$

Additionally, to ensure effective and homogeneous mixture of the starch composite, the mixing index of the screw extruder was computed from the empirical relationship in Equation 20.3.

$$M = \frac{S_o^2 - S_r^2}{S_o^2 - S_r^2} \quad (20.3)$$

where:

M is the mixing index, which ranges from 0 to 1 during the course of the mixing operation

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$S_r^2 \gg 0$ shows that the number of particles is very large

S_o^2 is the product of individual ingredients of the composite in the matrix

The intermediate values between S_r^2 and S_o^2 were used to show the progress of mixing. The subscripts o and r denote the initial and the random values of S^2 .

20.2.3 RATE OF MIXING OF THE STARCH COMPOSITES

The rate of mixing of the starch composite, which is based on the mixing index of the screw extruder, was evaluated from Equation 20.4. It is important to note that the mixing index is related to the rate of mixing at any time, under constant working conditions such as in a well-designed mixer working at constant speed, like the one under investigation, and this is proportional to the extent of mixing remaining to be accomplished at the time considered.

$$dM/dt = K[(1 - (M))] \quad (20.4)$$

where:

(M) is the mixing index

K is a constant

These are obtained from the experiment.

Integrating Equation 20.4 from $t = 0$ to $t = t$ during which (M) varies from 0 to (M) . The exponential relationship between the mixing index and the time of the mixing operation is expressed in Equation 20.5.

$$\begin{aligned} (1 - (M)) &= e^{-Kt} \\ (M) &= 1 - e^{-Kt} \end{aligned} \quad (20.5)$$

This exponential relationship is very important in the experimental investigations of the mixing index of composite, as in the present study. The constant K was related to the screw extruder and to the mixing conditions; and it was used to predict the times required to attain a given degree of mixing in the machine.

20.2.4 MODELING AND OPTIMIZATION OF THE STARCH COMPOSITE OUTPUT

RSM was applied to determine the optimum conditions of process variables for the output/1000 g of starch composite. The process variables considered in this studies were the diameter of the holes in the breaker plate ($X_1 = \emptyset$, 5–7 mm), concentration of glycerol in the composite ($X_2 = G$, 45%–55% w/v) and the concentration of the nanoparticles ($X_3 = N$, 0–2 mm), while the dependent response variable chosen was the output/1000 g of the starch composite. A Box–Behnken design (BBD) including 17 experiments formed by 5 central points was employed. Coded values corresponding

TABLE 20.1

Combination of Extrusion Process Variables and Output of the Starch Composite for Response Variable

Treatment ^a	Process Variables			Response Variable
	Ø of Breaker Plate (mm)	Glycerol Conc. (% w/v)	Conc. of Nanoparticles (% w/v)	Output/1000 g of Composite
1	6	55	2	0.000
2	7	50	2	0.402
3	6	50	1	0.000
4	5	45	1	0.448
5	5	50	2	0.502
6	6	50	1	0.000
7	7	45	1	0.359
8	6	45	2	0.000
9	6	50	1	0.000
10	6	50	1	0.000
11	6	50	1	0.000
12	6	55	0	0.000
13	7	50	0	0.394
14	6	45	0	0.000
15	7	55	1	0.442
16	5	55	1	0.552
17	5	50	0	0.493

^a Does not correspond to the order of processing.

to actual values of each variable and BBD are shown in Table 20.1, which shows individual experiments carried out in a random order (Vargas-Lopez et al., 1990). The quadratic polynomial regression model was assumed for predicting (Y) response variable. The model of the form expressed in Equation 20.6 was developed to describe the response (Y) surface:

$$\begin{aligned} Y &= \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 \\ &+ \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 \\ &+ \beta_{22} X_2^2 + \beta_{33} X_3^2 \end{aligned} \quad (20.6)$$

where:

Y is the value of the considered experimental predicted response variable (output of starch composite per 1000 g)

X_1 , X_2 , and X_3 are the values of $\emptyset$, G , and N , respectively

β_0 is the constant value

β_1 , β_2 , and β_3 are linear coefficients

β_{12} , β_{13} , and β_{23} are the interaction coefficient

β_{11} , β_{22} , and β_{33} are quadratic coefficients

Applying the stepwise regression procedure, nonsignificant terms ($p \leq .05$) were deleted from the second-order polynomial and a new polynomial was recalculated to obtain

a predictive model for each variable (Milán-Carrillo et al., 2006; Khuri and Cornell, 1987). All statistical analyses were performed using the Design Expert Package (ver. 9.0.3.1).

It is expedient to note that during the optimization of food processes, usually several response variables describing the quality characteristics and performance measures of the systems are to be optimized. Some of these variables are to be maximized, while others are to be minimized. In many cases, these responses are competing, that is, improving one response may have an opposite effect on another one, which further complicates the situation (Jahani et al., 2008). Several approaches have been used to tackle this problem. One of the most common is the conventional graphical method that superimposes the contour diagrams of the different response variables, whereas a second approach solving the problem of multiple responses uses a desirability function that combines all the responses into one measurement.

The conventional graphical method (CGM) is a relatively straightforward approach for optimizing several responses, and it works well when there are only a few process variables. CGM was used as an optimization technique to obtain maximum output/1000 g of the starch composite. Predictive models were used to represent graphically the system. Contour plots for each of the response variables were used applying superimposed surface methodology, to obtain a contour plot for observation and selection the zone of optimization of diameter of the holes in the breaker plate, the concentrations of glycerol, and the nanoparticles in the composite. To perform this operation, the Design Expert Package (ver. 9.0.3.1) was also used.

The fitted model can be evaluated at any point $X = (X_1, X_2, X_3)$ of the experimental zone and as a result two values were predicted for each model, namely $\hat{Y}_1(X)$, $\hat{Y}_2(X)$, and $\hat{Y}_3(X)$. Then each $\hat{Y}_i(x)$ s transformed into a value $d_i(x)$, which varies between 0 and 1, and measures the degree of desirability of the response with reference to the optimum value intended to be reached. In this research, we wanted all response variables to be as high as possible. Thus, the transformation is expressed in Equation 20.7.

$$d_i(x) = \begin{cases} 0 & \text{if } \bar{Y}_i(x) \leq Y_{i*} \\ \frac{\bar{Y}_i(x) - Y_{i*}}{Y_i^* - Y_{i*}} & \text{if } Y_{i*} \leq \bar{Y}_i(x) \leq Y_i^* \\ 1 & \text{if } \bar{Y}_i(x) \geq Y_i^* \end{cases} \quad (20.7)$$

where:

$d_i(x)$ is the value of the desirability of the i th response variable

$\bar{Y}_i(x)$ is the estimated response variable

Y_i^* is the maximum acceptable value of the i th response variable

Y_{i*} is the minimum acceptable value of the i th response variable

20.3 RESULTS AND DISCUSSION

20.3.1 MIXING INDEX AND RATE OF THE STARCH COMPOSITE EXTRUDER

The effects of the resident time of machine operation on the mixing index at different concentrations of glycerol and nanoparticles are shown in Figure 20.2. Decay trends were observed as the mixing index increases with an increase in the resident time between 45% and 55% w/v of the glycerol concentration and a decrease in the output per 1000 g of the starch composite through both the 5 and 7 mm diameters of the hole in the breaker plate. The highest mixing index was observed for the 45% w/v, and the lowest for the 55% w/v of the glycerol concentration in the starch composites. In each case, a higher mixing index was observed when the die head was turned to accommodate the 5 mm diameter of the holes in the breaker plate. Similarly, a lower mixing index was observed when the output was collected through the 7 mm diameter of the holes in the breaker plate. Although the force acting on the breaker plate was uniform as the speed of the rotating shaft was never altered throughout the machine operation, the difference in the diameter of the holes in the plate ensures variation in the output pressure, and hence the associated differences in the output of the composite at the die end of the extruder. In a related research, Connelly and Kokini (2007) examined the mixing ability of single- and twin-screw mixers using 2D finite element method simulation with particle tracking and stated the output increases with an increase in the pressure attended from the varying speeds of the shafts in both the twin- and the single-screw extruders. Additionally, Sau and Jana (2004) studied the effects of chaotic mixer design and operating conditions on the morphology development in immiscible polymer systems, and corroborated that operating conditions such as variable speed, pressures, and extruder geometry drastically affect the output at the die end of the extruder. Consequently, with an increase in the resident time of machine operation, the mixing indices at the various glycerol concentrations of the screw extruder with 5 mm

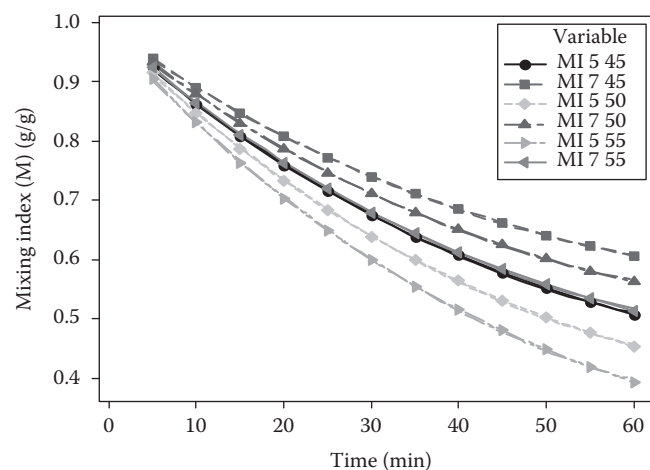


FIGURE 20.2 Effects of the resident time on the mixing index at different concentration of glycerol and nanoparticles.

diameter of the holes in the breaker plate are higher than those with 7 mm diameter of the hole. For instance, when the mixing time was 5 min, the probabilities of obtaining maximal mixing index for 45% of glycerol concentration in the single screw extruder were 97% and 95% at the 5 and 7 mm diameters, respectively, while the optimal mixing of the composite containing 50% glycerol concentration was 90% and 93% at the 5 and 7 mm diameter of the holes in the breaker plate. It further reveals that the extruder operating at the 5 mm diameter of the holes in the breaker plate has higher mixing index, and hence efficiency than the one operating at the 7 mm diameter of the hole.

The effects of the rate of composite mixing on the amount of unmixed products in the extruder, at 1% and 2% concentration of zinc oxide nanoparticles, are shown in Figure 20.3. The amount of unmixed composite in the extruder was found to decrease exponentially with an increase in the mixing rate. This is probably because of the decrease in the mixing index of the starch composite extruder with an increase in the diameter of the holes in the breaker plate throughout the period of machine operation. Additionally, only 5% of the amount of the composite fed into the screw extruder was unmixed in the first 10 min. But, the amount of the unmixed composite increased with time for 1 h, thereby decreasing the ratio of the mixing index and the resident time of machine operation. Theoretically, the mixing rate in the metering region is higher than those in other regions for single-screw extruder due to the greater shear rates experienced (Fadéyibi et al., 2014). It is clear that the mixing index distributions in the screw extruder, at the 45% are similar to those at the 50% glycerol concentration. Also, the data in Figure 20.3 were used to generate the expressions in Equations 20.8 through 20.13. The mixing rate expressions of the extruder revealed that the composite containing 55% glycerol concentration at 5 mm diameter of breaker plate has the highest mixing rate

constant of magnitude 0.527. The lowest constant value of the mixing rate obtained for the composite containing 45% glycerol concentration at 5 mm diameter of the holes breaker plate was of the order of magnitude 0.358, while other constant values occurred between these two values at higher coefficients of determination. This implies that the screw extruder has greater mixing rate and efficiency at the 55% glycerol concentration than at the other two concentrations of glycerol in the starch composite. Consequently, the larger the value of the mixing rate efficiency, the lower the amount of the unmixed composite in the barrel of the screw extruder.

$$dM/dt = 0.439e^{-10.6(1-M)} \quad R^2 = 0.906(5\phi 45) \quad (20.8)$$

$$dM/dt = 0.358e^{-10.6(1-M)} \quad R^2 = 0.907(7\phi 45) \quad (20.9)$$

$$dM/dt = 0.482e^{-10.7(1-M)} \quad R^2 = 0.905(5\phi 50) \quad (20.10)$$

$$dM/dt = 0.394e^{-10.7(1-M)} \quad R^2 = 0.905(7\phi 50) \quad (20.11)$$

$$dM/dt = 0.527e^{-10.7(1-M)} \quad R^2 = 0.902(5\phi 55) \quad (20.12)$$

$$dM/dt = 0.433e^{-10.6(1-M)} \quad R^2 = 0.906(7\phi 55) \quad (20.13)$$

20.3.2 EFFECT OF THE DIAMETER OF THE HOLES IN THE BREAKER PLATE ON THE COMPOSITE OUTPUT

The variations of the output of starch composite with the diameter of the holes in the breaker plate for the single-screw extruder are presented in Tables 20.2 through 20.4. The mixed output of the starch composite from the machine was higher at the 5 mm diameter of the holes in the breaker plate, and lower at its 7 mm counterpart. Moreover, for the two diameters considered, the output was higher at the end of the 1 h machine operation, with an almost negligible difference among the outputs obtained. This is probably because of the pressure increase accompanying greater force at lower area of cross section or diameter of the holes in the breaker plate. Additionally, the relationship between the predicted output of starch composite per 1000 g and the responsible or actual output of the screw extruder can be seen in Figure 20.4. It shows the slight rise in the output of the starch composite at the initial mixing time, because the material in the extruding channel is prone to stretch because of its very small area. Lindt and Ghosh (1992) studied the fluid mechanics of the formation of polymer blends and suggested that the degree of mixing increases with the generation of interfacial area between the individual material components and with the residence time. Thus, the shear strain of the mixed composite will play an important role in the generation of this interfacial area. As stated above, the output obtained from each of the diameters of the holes in the breaker plate varies with its associated pressure through the plate. Therefore, a simple way to quantify the output of an extruder can be the average pressure generated at the plate (Pinto and Tadmor, 1970).

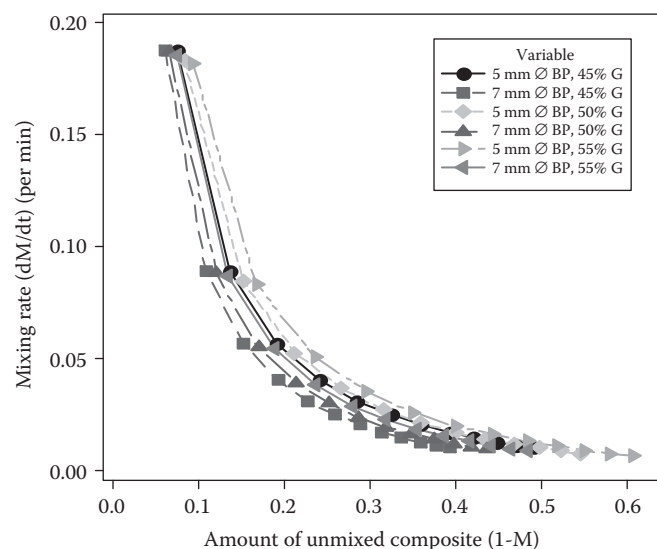


FIGURE 20.3 The relationship between the amount of the unmixed composite and the mixing rate.

TABLE 20.2

Analyses of the Mixing Index and Rate of the Starch Composite at 45% (w/v) Glycerol Concentration

Output of Starch at 45% (w/v) Glycerol Per 1000 g of Composite Fed in the Hopper													Mixing Rate Analyses			
T	5 mm Ø BP			7 mm Ø BP			Mixing Index Analyses						$d M_5/dt$	$d M_7/dt$	$(1-M_1)$	$(1-M_1)$
min	0	1	2	0	1	2	$(S)_5^2$	$(S)_7^2$	$(S_0)^2$	$(S_r)^2$	M_5	M_7	min ⁻¹	min ⁻¹	g/g	g/g
5	0.068	0.069	0.070	0.055	0.0552	0.056	0.00068	0.0005	0.009	0.000	0.924	0.9392	0.1848	0.1878	0.076	0.061
10	0.123	0.124	0.126	0.098	0.0994	0.100	0.00123	0.0010	0.009	0.000	0.863	0.8906	0.0863	0.0891	0.137	0.109
15	0.172	0.174	0.176	0.138	0.1392	0.141	0.00172	0.0014	0.009	0.000	0.809	0.8468	0.0539	0.0565	0.191	0.153
20	0.217	0.219	0.221	0.173	0.1750	0.177	0.00217	0.0017	0.009	0.000	0.759	0.8074	0.0380	0.0404	0.241	0.193
25	0.257	0.259	0.262	0.205	0.2073	0.209	0.00257	0.0021	0.009	0.000	0.715	0.7720	0.0286	0.0309	0.285	0.228
30	0.292	0.295	0.298	0.234	0.2363	0.239	0.00292	0.0023	0.009	0.000	0.675	0.7401	0.0225	0.0247	0.325	0.260
35	0.325	0.328	0.331	0.260	0.2624	0.265	0.00325	0.0026	0.009	0.000	0.639	0.7114	0.0183	0.0203	0.361	0.289
40	0.354	0.357	0.361	0.283	0.2858	0.289	0.00354	0.0028	0.009	0.000	0.607	0.6855	0.0152	0.0171	0.393	0.314
45	0.380	0.384	0.388	0.304	0.3070	0.310	0.00380	0.0030	0.009	0.000	0.578	0.6623	0.0128	0.0147	0.422	0.338
50	0.403	0.408	0.412	0.323	0.3260	0.329	0.00403	0.0032	0.009	0.000	0.552	0.6414	0.0110	0.0128	0.448	0.359
55	0.425	0.429	0.433	0.340	0.3431	0.347	0.00425	0.0034	0.009	0.000	0.528	0.6225	0.0096	0.0113	0.472	0.377
60	0.444	0.448	0.453	0.355	0.3585	0.362	0.00444	0.0035	0.009	0.000	0.507	0.6056	0.0084	0.0101	0.493	0.394

BP, breaker plate; M_5 , mixing index at 5 mm Ø BP; M_7 , mixing index at 7 mm Ø BP; dM_5/dt , mixing rate at 5 mm Ø; and dM_7/dt , mixing rate at 7 mm Ø.

TABLE 20.3

Analyses of the Mixing Index and Rate of the Starch Composite at 50% (w/v) Glycerol Concentration

Output of Starch at 50% (w/v) Glycerol Per 1000 g of Composite Fed in the Hopper														Mixing Rate Analyses			
T	5 mm Diameter			7 mm Diameter			Mixing Index Analyses						$d M_5/dt$	$d M_7/dt$	$(1-M_1)$	$(1-M_1)$	
min	0	1	2	0	1	2	$(S)_5^2$	$(S)_7^2$	$(S_0)^2$	$(S_r)^2$	M_5	M_7	min ⁻¹	min ⁻¹	g/g	g/g	
5	0.076	0.077	0.077	0.061	0.0613	0.062	0.00076	0.0006	0.009	0.000	0.916	0.9325	0.1831	0.1865	0.084	0.067	
10	0.137	0.138	0.139	0.109	0.1104	0.111	0.00137	0.0011	0.009	0.000	0.848	0.8786	0.0848	0.0879	0.152	0.121	
15	0.191	0.193	0.195	0.153	0.1545	0.156	0.00191	0.0015	0.009	0.000	0.787	0.8300	0.0525	0.0553	0.213	0.170	
20	0.240	0.243	0.245	0.192	0.1943	0.196	0.00240	0.0019	0.009	0.000	0.733	0.7863	0.0366	0.0393	0.267	0.214	
25	0.285	0.288	0.290	0.228	0.2301	0.232	0.00285	0.0023	0.009	0.000	0.684	0.7469	0.0273	0.0299	0.316	0.253	
30	0.325	0.328	0.331	0.260	0.2622	0.265	0.00325	0.0026	0.009	0.000	0.639	0.7115	0.0213	0.0237	0.361	0.288	
35	0.360	0.364	0.368	0.288	0.2912	0.294	0.00360	0.0029	0.009	0.000	0.600	0.6796	0.0171	0.0194	0.400	0.320	
40	0.393	0.397	0.401	0.314	0.3173	0.320	0.00393	0.0031	0.009	0.000	0.564	0.6510	0.0141	0.0163	0.436	0.349	
45	0.422	0.426	0.430	0.337	0.3408	0.344	0.00422	0.0034	0.009	0.000	0.531	0.6251	0.0118	0.0139	0.469	0.375	
50	0.448	0.452	0.457	0.358	0.3619	0.365	0.00448	0.0036	0.009	0.000	0.502	0.6019	0.0100	0.0120	0.498	0.398	
55	0.471	0.476	0.481	0.377	0.3809	0.385	0.00471	0.0038	0.009	0.000	0.476	0.5810	0.0087	0.0106	0.524	0.419	
60	0.493	0.497	0.502	0.394	0.3980	0.402	0.00493	0.0039	0.009	0.000	0.453	0.5622	0.0075	0.0094	0.547	0.438	

BP, breaker plate; M_5 , mixing index at 5 mm Ø BP; M_7 , mixing index at 7 mm Ø BP; dM_5/dt , mixing rate at 5 mm Ø; and dM_7/dt , mixing rate at 7 mm Ø.

The desirability method described by De la Vara and Domínguez (2002) was applied to find the best combination of extrusion process variables (diameter of the holes in the breaker plate [Ø], concentration of the glycerol [G], and concentration of nanoparticles [N]) that will give the optimum value for the output of the starch composite. Figure 20.5 shows the individual desirability for response variables (Output/1000 g of

starch composite) and the global desirability for the optimized extruded process variables. The predicted output of the starch composite with increasing interaction was 0.530/1000 g, and this occurs in all the concentrations of the glycerol and the diameters of the holes in the breaker plate. The individual and the global desirabilities are well in agreement with each other for maximum output of the starch composite (0.530/1000 g)

TABLE 20.4
Analyses of the Mixing Index and Rate of the Starch Composite at 55% (w/v) Glycerol Concentration

Output of Starch at 55% (w/v) Glycerol Per 1000 g of Composite Fed in the Hopper										Mixing Rate Analyses							
T	5 mm Diameter			7 mm Diameter			Mixing Index Analyses						$d M_s/dt$ min^{-1}	$d M_r/dt$ min^{-1}	$(1-M_r)$ g/g	$(1-M_s)$ g/g	
min	0	1	2	0	1	2	$(S_s)^2$	$(S_r)^2$	$(S_0)^2$	$(S_s)^2$	$(S_r)^2$	M_s	M_r				
5	0.084	0.085	0.086	0.067	0.0681	0.069	0.00084	0.0007	0.009	0.000	0.000	0.906	0.9251	0.1813	0.1850	0.094	0.075
10	0.152	0.153	0.155	0.121	0.1225	0.124	0.00152	0.0012	0.009	0.000	0.000	0.832	0.8652	0.0832	0.0865	0.168	0.135
15	0.212	0.214	0.217	0.17	0.1715	0.173	0.00212	0.0017	0.009	0.000	0.000	0.764	0.8113	0.0509	0.0541	0.236	0.189
20	0.267	0.27	0.272	0.214	0.2157	0.218	0.00267	0.0021	0.009	0.000	0.000	0.703	0.7628	0.0352	0.0381	0.297	0.237
25	0.316	0.319	0.322	0.253	0.2554	0.258	0.00316	0.0025	0.009	0.000	0.000	0.649	0.7191	0.0260	0.0288	0.351	0.281
30	0.360	0.364	0.367	0.288	0.2911	0.294	0.00360	0.0029	0.009	0.000	0.000	0.600	0.6798	0.0200	0.0227	0.400	0.320
35	0.400	0.404	0.408	0.32	0.3232	0.326	0.00400	0.0032	0.009	0.000	0.000	0.555	0.6444	0.0159	0.0184	0.445	0.356
40	0.436	0.44	0.445	0.349	0.3522	0.356	0.00436	0.0035	0.009	0.000	0.000	0.516	0.6126	0.0129	0.0153	0.484	0.387
45	0.468	0.473	0.477	0.374	0.3782	0.382	0.00468	0.0037	0.009	0.000	0.000	0.480	0.5839	0.0107	0.0130	0.520	0.416
50	0.497	0.502	0.507	0.398	0.4017	0.406	0.00497	0.0040	0.009	0.000	0.000	0.448	0.5581	0.0090	0.0112	0.552	0.442
55	0.523	0.528	0.534	0.419	0.4228	0.427	0.00523	0.0042	0.009	0.000	0.000	0.419	0.5349	0.0076	0.0097	0.581	0.465
60	0.547	0.552	0.558	0.437	0.4418	0.446	0.00547	0.0044	0.009	0.000	0.000	0.393	0.5140	0.0065	0.0086	0.607	0.486

BP, breaker plate; M_s , mixing index at 5 mm Ø BP; M_r , mixing index at 7 mm Ø BP; $d M_s/dt$, mixing rate at 5 mm Ø; and $d M_r/dt$, mixing rate at 7 mm Ø.

BP, breaker plate; M_s , mixing index at 5 mm Ø BP; M_r , mixing index at 7 mm Ø BP; dM_s/dt , mixing rate at 5 mm Ø; and dM_r/dt , mixing rate at 7 mm Ø.

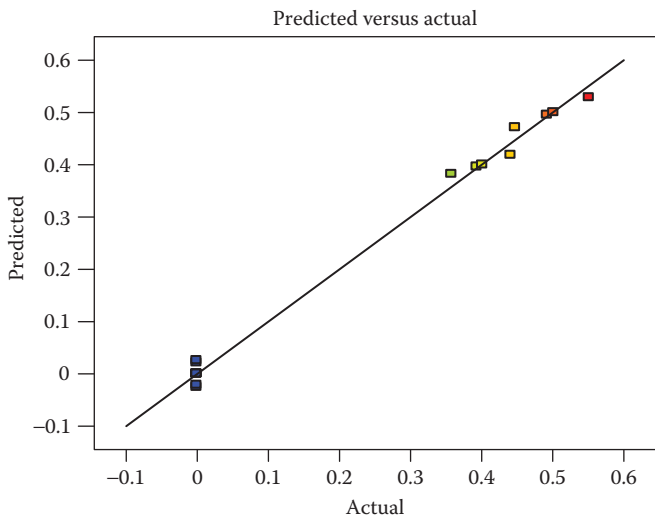


FIGURE 20.4 Relationship between the predicted and actual output/1000 g of composite.

at 5 mm diameter of the holes in the breaker plate and 55% w/v of the glycerol concentration. In a related research, Milán-Carrillo et al. (2012) optimized the extrusion process for producing high antioxidant instant amaranth (*Amaranthus*

hypochondriacus L.) flour using RSM, and determined the best combination of the extrusion process variables for the production of extruded amaranth flour to elaborate a beverage with high antioxidant capacity and acceptability. Thus, the optimum values of the process combinations obtained can well be selected for use during the assessment of the performance of the single-screw extruder.

20.3.3 OPTIMIZATION AND MODELING OF THE OUTPUT OF THE STARCH COMPOSITE

The optimization and modeling of the concentration of nanoparticles and the diameters of the holes in the breaker plate for the single-screw extruder in order to predict the response of the system in terms of the output of the starch composite were undertaken in this study. Figure 20.6 shows the effect of the glycerol concentration and diameter of the holes in the breaker plate on the output of starch composite, which presents the output of the starch composite affecting each of the three criteria for the three factors considered. Since it is easier to assess the value of the predictions, the optimization methodology was tested for the identification of the best combination of the concentration of nanoparticles and the diameters of the holes in the breaker plate, as defined for each

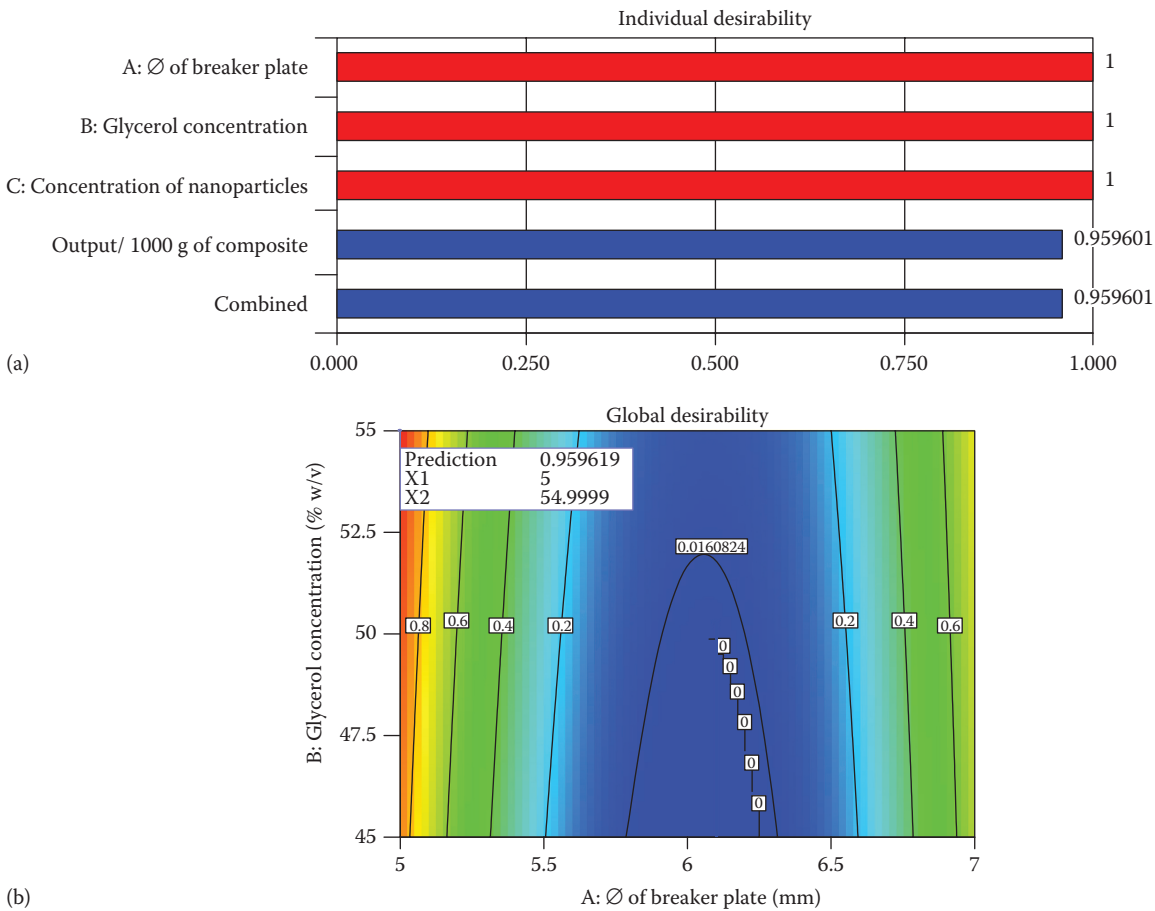


FIGURE 20.5 (a) Individual desirability for response variables (Output/1000 g of starch composite) (0.9596). (b) Global desirability for the optimized extruded process variables (0.9596).

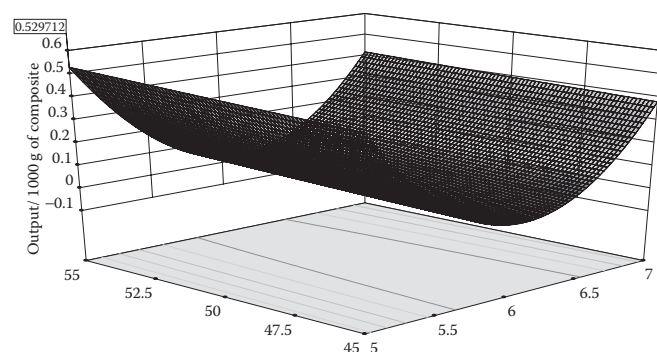


FIGURE 20.6 The effect of glycerol concentration and diameter of breaker plate on the output of starch composites: (a) Ø of breaker plate (mm) and (b) glycerol concentration (% w/v).

individual factor, to maximize the output of the starch composite. Consequently, all the factors were considered simultaneously to compare the outputs of the starch composite for the best combination of the concentration of nanoparticles and the diameters of the holes in the breaker plate obtained when the various factors were considered individually. Figure 20.6 also depicts the actual output of the starch composite for the best factor when the output was maximized. A concentration of glycerol of 55% and a 5 mm diameter of the holes in the breaker plate actually maximize the output of the starch composite, up to 0.53/1000 g, with a desirability of 0.96. The higher output of the starch composite obtained in this investigation follows the guidance provided by Rauwendaal (1986)

when his analytical method was applied to the simultaneous optimization of the channel depth and helix angle during melt conveying for a maximum output in the screw extruder. In the case of ensuring adequate mixing, maximum output, and efficiency, the starch composite must be made of 55% glycerol concentration, 1% zinc oxide nanoparticles, and a 5 mm diameter of the holes in the breaker plate.

A model for the agglomerate dispersion of the starch composite in single-screw extruder is also proposed. The proposed model predicts the morphology of such systems, by computing the output of the starch composite in the distribution of the population of agglomerates present in the matrix. The model is quite general and can be adapted to different type of starch, nanoparticles, and the diameter of the holes in the breaker plate as well as for different type of plasticizer, such as urea, water, and polyethylene glycol. The analysis in this study shows that the output of the starch composite per 1000 g depends not only on the diameter of the holes in the breaker plate but also on the concentrations of the glycerol and the nanoparticles. In view of this therefore, with a fair knowledge of the resident time, the throughput of the screw extruder, at each stage of the mixing operation, can be computed from the model as well. The results corroborates the fact that a starch composite containing solid additives tends to be homogenous with time. Such homogeneity is highly dependent on the operating conditions. The model equation has the predicted coefficient of determination of 0.9198, reasonably higher and in agreement with the adjusted coefficient of determination of 0.9885. Furthermore, Table 20.5 shows showed that the output of the starch composite per 1000 g was significantly dependent on the linear terms

TABLE 20.5
Analyses of Variance of the Output of the Starch Composite

Source of Variation	Sum of Squares	df	Mean Square	F Value	p-Value Prob > F	Significant
Model	0.87792	9	0.097546	154.44	3.3E-07	Significant
A-Ø of Breaker Plate	0.01987	1	0.01987	31.459	0.00081	
B-Glycerol Concentration	0.00439	1	0.004385	6.9427	0.03367	
C-Concentration of Nanoparticles	3.6E-05	1	3.61E-05	0.0572	0.81784	
AB	0.00011	1	0.000107	0.1696	0.69279	
AC	2.5E-07	1	2.5E-07	0.0004	0.98468	
BC	0	1	0	0	1	
A ²	0.84852	1	0.848516	1343.4	2.9E-09	
B ²	5.7E-06	1	5.69E-06	0.009	0.92704	
C ²	5.7E-06	1	5.69E-06	0.009	0.92704	
Residual	0.00442	7	0.000632			
Lack of fit	0.00442	3	0.001474			
Pure error	0	4	0			
Correlation total	0.88234	16				

CV, coefficient of variation = 11.

of the diameter of the breaker plate ($\emptyset BP$, $p < .01$), glycerol concentration (GL, $p < .05$) and concentration of nanoparticles (NP, $p < .01$), as presented in Equation 20.14.

$$\begin{aligned} \text{Output} = & 1.02 \times 10^{-16} - 0.04984 \emptyset BP + 0.023413 GL \\ & + 0.002125 NP - 0.00518 \emptyset BP \cdot GL \\ & - 0.00025 \emptyset BP \cdot NP - 4.6 \times 10^{-20} GL \cdot NP \\ & + 0.448913 \emptyset BP^2 + 0.001162 GL^2 - 0.00116 NP^2 \end{aligned} \quad (20.14)$$

where:

$\emptyset BP$ is the diameter of the holes in the breaker plate

GL is the glycerol concentration

NP is the concentration of nanoparticles

20.4 CONCLUSIONS

The mixing index of a starch composite extruder was determined for cassava starch, glycerol, and zinc oxide nanoparticles in a single matrix and of different proportions. Two diameters, namely 5 and 7 mm, which are an integral part of the extruder at its die end, were used to access the performance of the extruder with respect to the output/1000 g of the starch composite. The mixing index and rate of the extruder were computed, and the amount of the unmixed product in the barrel containing the screw conveyor was also accessed. Decay trends were observed as the mixing index increases with an increase in the resident time in the range of 45%–55% w/v glycerol concentration and a decrease in the output per 1000 g of the starch composite through both the 5 and 7 mm diameters of the hole of the breaker plate. The amount of the unmixed composite in the extruder decreased exponentially with an increase in the mixing rate. The composite containing 55% glycerol and 1% zinc oxide nanoparticles at 5 mm diameter of holes in the breaker plate has the highest mixing rate constant with magnitude of 0.527, which is an indication of higher performance in terms of extruder output at any given time.

Additionally, the process parameters were optimized, and the desirability for the best combination of the extrusion process variables (diameter of the holes in the breaker plate [$\emptyset BP$], concentration of the glycerol [GL], and concentration of nanoparticles [NP]) that will give the optimum value for the output of the starch composite was determined. A quadratic model equation was generated to relate all the factors as extrusion process variables, and the output/1000 g of the starch composite as the response variable. The model was adequately fitted with the predicted coefficient of determination of 0.9198, reasonably higher and in agreement with the adjusted coefficient of determination of 0.9885. The combination with the concentrations of 55% GL, 1% NP, and a 5 mm $\emptyset BP$ maximizes the output of the starch composite, up to approximately 0.53/1000 g, with a desirability of 0.96. Thus,

the optimum values of the process combinations obtained can as well be selected for use in the assessment of the performance of the single-screw extruder.

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Author Query Sheet

Chapter No.: 20

Query No.	Queries	Response
AQ 1	Please clarify “This encapsulates.”	
AQ 2	Please check the edits made to the sentence “The chaotic mixing...”.	
AQ 3	Please check the edits made to the sentence here is OK.	
AQ 4	The sense of the sentence “It is clear that the mixing...” seems unclear; please check.	
AQ 5	Equation number 20.6 has been changed to 20.14 to maintain the sequential order. Please check.	
AQ 6	Please provide the editors name and page range for Potente et al. (1994).	