

# Dry Matter Loss During Nixtamalization of a White Corn Hybrid: Impact of Processing Parameters

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## ABSTRACT

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Nixtamalization is the primary step in the production of products such as corn chips, tortilla chips, tacos, and corn tortillas. The process involves cooking and steeping of corn in lime and excess water to produce nixtamal. Commercial nixtamalization results in 5–14% corn solids loss in the liquid generated during cooking-steeping and washing. Loss of corn solids not only causes economic loss to corn processors but also creates costly waste and wastewater disposal problems. Empirical results show that, besides corn kernel characteristics, processing parameters are critical variables influencing corn solids loss and effluent pH during nixtamali-

zation. This work was designed to systematically study the impact of processing parameters on corn dry matter loss and effluent pH generated during nixtamalization by using response surface methodology. Corn cooking temperature and lime concentration were more critical factors influencing corn solid loss than were cooking and steeping time. In the ranges studied, total dry matter loss increased only up to ≈8 hr of steeping and then leveled off. By optimizing the nixtamalization protocol, effluent dry matter loss can be minimized.

Alkaline cooking of corn with lime, traditionally called nixtamalization, is the primary processing step during manufacture of several corn products such as corn chips, tortilla chips, corn tortillas, and taco shells. Even with growing popularity of these corn products, little improvement has been made in this ancient corn processing method practiced by the Aztecs. The basic process begins by cooking whole corn in water with lime and steeping the cooked corn for 12–16 hr in large tanks. The steeped corn is *nixtamal* and the cook-steep liquid, rich in corn solids, is *nejayote*. This pre-Columbian alkaline cooking technique softens the pericarp and allows the endosperm to absorb water, thus facilitating its milling. Nixtamal is washed to remove loose pieces of pericarp and stone-ground to produce masa. Masa or corn dough is used to produce tortillas, taco shells, tostadas, tamales, and snacks such as corn chips and tortilla chips. Traditionally, hard white corn is used for tortilla making in Mexico as it results in a “superior” white tortilla and relatively less corn solids loss as compared with softer corn types (Martinez-Herrera and Lachance 1979, Jackson et al 1988).

Alkaline cooking, steeping, and washing of corn causes a partitioning of corn solids between masa and wastewater (Jackson 1988). A corn nixtamalization facility processing 200 U.S. tons of corn per day uses over 50 gallons of water per minute and generates nearly the equivalent amount of alkaline wastewater in a 24-hr period (M. Wagoner, C. J. Schneider Engineering, Omaha, NE, *personal communication*). Loss of corn solids (i.e., yield loss during nixtamalization) and cost of effluent processing are considerable. In a commercial operation, corn solids loss has been estimated at 5–14% depending on the type of corn (Katz et al 1974, Khan et al 1982). Corn solids lost during nixtamalization are 75.6% nonstarch polysaccharides, 11.6% starch, 1.4% protein, and contain high levels of calcium (Katz et al 1974). In a systematic evaluation of corn dry matter loss (DML) in two commercial plants where corn was steeped overnight, Pflugfelder et al (1988) reported 8.5–12.5% process loss. They also reported that nejayote solids contained ≈64% nonstarch polysaccharides (primarily pericarp fiber), 20% starch, and 1.4% protein.

The effluents generated during alkaline cooking of corn (*nejayote*) are potential pollutants because of their composition and characteristics. In large commercial establishments, where efforts are focused on expediting the nixtamalization process and mini-

mizing steeping times, corn is usually overcooked (high-temperature short-time cooking) and quenched with water to rapidly reduce corn temperatures. This approach shortens process times. However, it increases corn DML and water use. The commercial, so-called instant masa process, which produces a dry masa flour, also uses batch nixtamalization or continuous cooking, resulting in some of the same waste and efficiency concerns.

The magnitude and composition of corn solid loss at each step during nixtamalization is dependent on corn quality, kernel characteristics, and processing conditions (Jackson et al 1988, Pflugfelder et al 1988). For a given corn hybrid, process variables such as cook temperature, cook time, steep time, and concentration of lime appear to be the primary factors influencing DML during nixtamalization. It appears possible to study and appropriately vary these process variables in various combinations so that corn is nixtamalized for an acceptable masa texture. Our research was undertaken to study the impact of these process variables (cook temperature, cook time, steep time, and concentration of lime) on DML during nixtamalization using response surface methodology (RSM). Such an approach can help in developing nixtamalization protocols whereby DML is minimized.

## MATERIALS AND METHODS

### Corn Sample

White corn, traditionally used for tortilla production, grown in the Sinaloa region of Mexico (1997 crop), was imported for the study. The corn was commercially cleaned at FlatWater Mills (Hastings, NE) and stored in a freezer (–18°C) before nixtamalization.

### Corn Characterization

A composite corn sample was collected for characterization from 38 bags. Corn (≈200 g) was removed from each bag taken for the nixtamalization experiments and combined to form the composite sample.

Moisture content of the composite sample was determined by Approved Method 44-15A (AACC 1995). A tangential abrasive dehulling device (TADD, model 4E-220, Venables Machine Works, Saskatoon, SK, Canada) was used to measure kernel hardness. TADD losses were the percent loss of kernel material after abrading 40 g of maize for 10 min while suctioning off abraded material as described by Reichert et al (1986).

The Stenvert Hardness test involved grinding 20 g of maize using a micro-hammermill (GlenMills model V, fitted with a 2-mm screen) at 360 rpm. The heights of soft endosperm and total ground material collected in the recovery tube, time to grind, reduced

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hammermill rpm at maximum grinding power, and quantity of hard endosperm recovered over a 425-mm sieve was measured (Pomeranz et al 1985).

The breakage susceptibility of the corn sample was determined by using a Wisconsin breakage tester (model 9/84, no. C0220, 1,800 rpm) as described by Watson and Herum (1986) and Pomeranz et al (1986). Stress-cracked and broken kernels were observed on a brightly lit box with the light source under the kernels. The grain samples were classified as undamaged, single stress cracked, multiple stress cracked, or checked-crazed as described by Thompson and Foster (1963).

Thousand-kernel weight was determined by hand-counting 100 whole kernels of each sample and multiplying the weight by 10. Floaters were determined as percent buoyant kernels immersed in a 31.3 Baume solution of sodium nitrate maintained at 60°C corresponding to a specific gravity of 1.275 (Peplinski et al 1989).

All the above tests were conducted in triplicate, average values of results along with standard deviations were reported. The results were interpreted as described by Shandera et al (1997).

### Nixtamalization Procedure

A standardized protocol was developed for corn nixtamalization that was uniformly followed for all experimental trials. Water (150 kg) was weighed accurately and pumped into a gas-fired cooking-steeping tank. Food grade lime Ca(OH)<sub>2</sub> (Mississippi Lime Co., Alton IL) was added to the tank and mixed using a wooden ladle until dispersed. The tank was covered with a lid and the lime solution was heated to the desired temperature. Corn (30 kg) was added into the tank. The corn was gently mixed to ensure a uniform temperature in the vessel. With the addition of corn to preheated water, the temperature of the cooking tank declined by ≈5–10°C. Heating was continued until the temperature of the corn-water lime mixture

recovered to the specified cooking temperature; cook time count-down was then started. Corn was cooked for the required time outlined in the experimental design. Tank temperature was maintained thermostatically within ±1°C during cooking.

Corn was cooked covered with a lid to reduce water vapor loss and minimize energy dissipation. The temperature also reached the desired cooking temperature more rapidly after addition of corn, and temperatures could be maintained with less fluctuation.

At the end of cooking, heating was discontinued, the lid of the cooking tank removed and steeping started. The corn was steeped for the required time as specified in the experimental design. Steeping was terminated by pumping the nejayote into large drums. To collect all the nejayote, nixtamal was allowed to drain for 30 min before washing. The weight and volume of collected nejayote was recorded. A representative sample of nejayote (≈8 L) was removed for subsequent analysis.

After draining the nejayote, nixtamal was washed twice using 50 L of water for each wash. Nixtamal was vigorously stirred in the wash water with a wooden paddle for ≈5 min. The wash water was removed by pumping it into large drums. To collect all the wash water, nixtamal was allowed to drain for another 30 min. The weight and volume of collected wash water was also recorded and a representative sample of ≈8 L was removed for analysis.

**TABLE I**  
Minimum and Maximum Ranges Selected for Four Nixtamalization Factors Generating the Central Composite Design

Factors	Units	Range	
		Minimum	Maximum
Cook temperature	°C	84.50	93.50
Cook time	min	26.25	48.75
Steep time	hr	4.00	12.00
Lime concentration (corn weight basis)	%	0.88	1.62

**TABLE II**  
Physical Characteristics of White Corn Used in Nixtamalization

Physical Characterization Test	Value	Standard Deviation
Wisconsin breakage	37.90%	1.11
TADD <sup>a</sup> index	58.78%	3.46
Stenvert hardness	68.33%	0.57
Floater percent	33.33%	2.31
1,000 kernel weight	317.7 g	4.05
Stress cracked broken kernels	6.73%	0.17

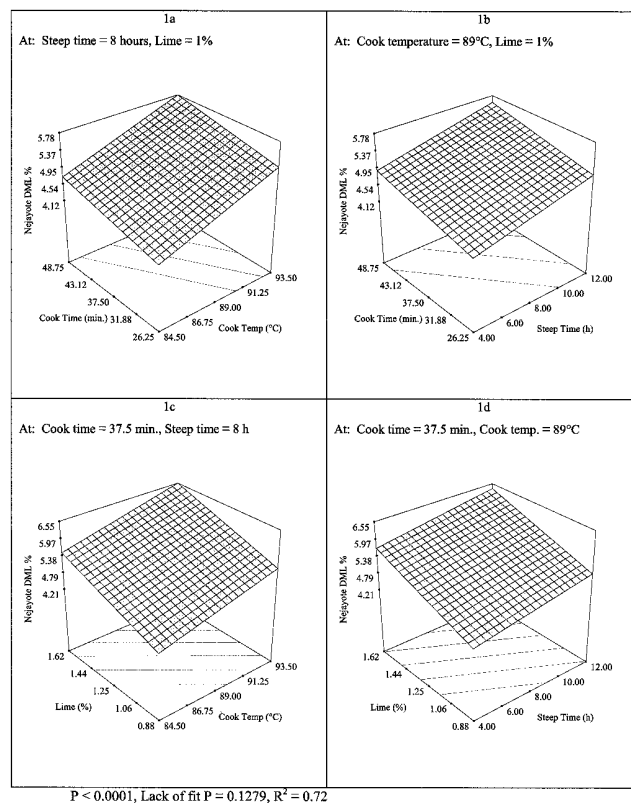
<sup>a</sup> Tangential abrasive dehulling device.

**TABLE III**  
Regression Coefficients (*R*) and Individual Significance (*P*) Values for Nejayote, Wash, and Total Dry Matter Loss (DML)

Nixtamalization Factors	Nejayote DML <sup>a</sup>		Wash DML <sup>a</sup>		Total DML <sup>b</sup>	
	<i>R</i>	<i>P</i>	<i>R</i>	<i>P</i>	<i>R</i>	<i>P</i>
Cook temperature (°C)	0.51	0.0003	0.18	0.0195	0.69	0.0002
Cook time (min)	0.28	0.0295	0.011	0.8782	0.29	0.0447
Steep time (hr)	0.34	0.0101	-0.07	0.3481	0.27	0.0577
Lime concentration (%)	0.62	<0.0001	0.39	<0.0001	1.01	<0.0001
[Lime concentration] <sup>2</sup>	...	...	...	...	-0.34	0.0275

<sup>a</sup> Represents a significant liner regression model.

<sup>b</sup> Represents a significant quadratic regression model.



**Fig. 1.** Response surface graphs for corn dry matter loss in nejayote.

## Nejayote and Wash Water Analysis

To estimate the solids content in nejayote and washwater, a representative sample of 8L was collected. Subsamples (200 mL each) were removed in duplicate into preweighed aluminum pans. The pans were dried in a forced-air oven maintained at 103°C for 12 hr. The total DML was calculated for 30 kg of corn and represented as %DML (w/v) in nejayote and washwater separately. The pH of nejayote and wash water was also recorded.

## Experimental Design

RSM was used as an experimental design to evaluate the impact of corn nixtamalization conditions on DML. RSM facilitated the study of multiple variables (cook temperature, cook time, lime concentration, and steep time) on DML. A central composite design (CCD) with 27 trials, six central points, one star point, and one axial point was used as the experimental design. The experimental design was generated and analysis was performed using Design Expert statistical software (Version 5, Stat-Ease Inc., Minneapolis, MN). Ranges of cook-steep variables described in Table I were selected based on preliminary nixtamalization trials and literature data.

## RESULTS AND DISCUSSION

### Corn Characterization

The results of various hardness and characterization tests of the white corn hybrid used in this study are represented in Table II. The corn sample was graded as medium hard (#2 hardness index class) as interpreted from the hardness index classification graph drawn between corn moisture and percent floaters. The composite corn sample had a moisture content of 10.6% (as-is, wb) and a test weight of 75.8 kg/hL.

### Nixtamalization DML in Nejayote

DML in nejayote from different cooking treatments exhibited a significant linear regression model ( $P < 0.0001$ , lack-of-fit  $P = 0.1279$ ,  $R^2 = 0.72$ ). This indicated that corn solid loss in nejayote

during nixtamalization was directly dependent on the processing factors tested. The slopes of the response surfaces generated for nejayote DML (Fig. 1) indicated that cook temperature and concentration of lime were the primary factors governing DML, whereas, cook time and steep time were of secondary importance. The observation is also supported by lower  $P$  values for regression coefficients of cook temperature and lime concentration as compared with cook time and steep time (Table III). This suggests that in a dynamic nixtamalization system, increasing lime concentration or cook temperature, has a greater adverse effect on DML accumulation rate as compared with increasing cook time or steep time.

According to the regression model, increasing cook temperature from 85 to 95°C when other variables are constant (30-min cook time, 1% lime, and 8-hr steep time) will increase DML from 4.31 to 5.25%. We observed that increasing lime concentration from 0.5 to 1.5% increased corn kernel rupture (particularly at high cook temperatures), thus increasing nejayote DML. Increases in steep time also linearly increased nejayote DML.

### Nixtamalization DML in Washwater

DML in washwater exhibited a significant linear regression model ( $P < 0.0002$ , lack-of-fit  $P = 0.1325$ ,  $R^2 = 0.72$ ), indicating a direct dependence on processing parameters. Cook temperature and concentration of lime were the most critical factors governing DML (Fig. 2., Table III). Corn DML decreased linearly with increasing steep time during washing. This suggests that during steeping partitioning of corn solids occurs between the solid (nixtamal) and the liquid phases (nejayote) so that more corn solids are released in nejayote and fewer are left to be washed and removed in the washwater.

### Total DML

Total DML in nejayote and washwater exhibited a quadratic regression model ( $P < 0.0003$ , lack-of-fit  $P = 0.0901$ ,  $R^2 = 0.90$ ) with processing parameters. Because of the variability associated with solids

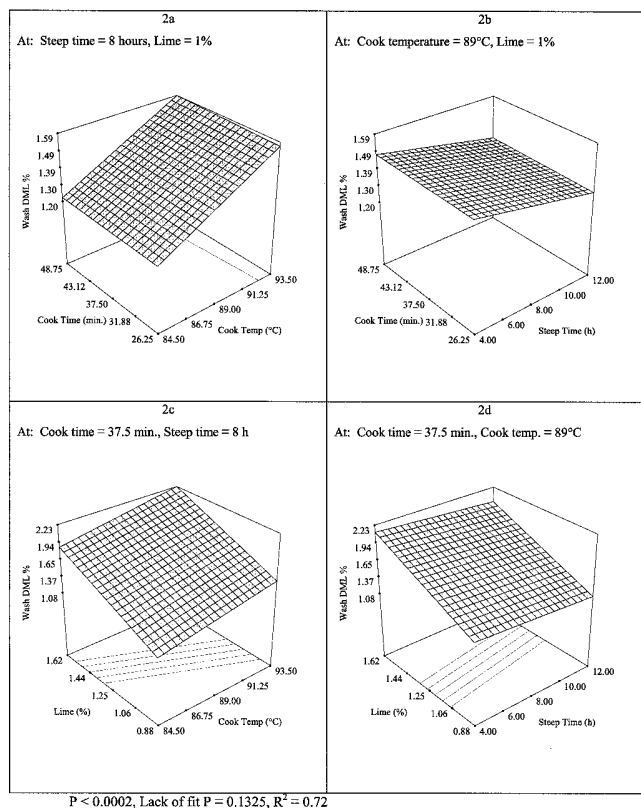


Fig. 2. Response surface graphs for corn dry matter loss in washwater.

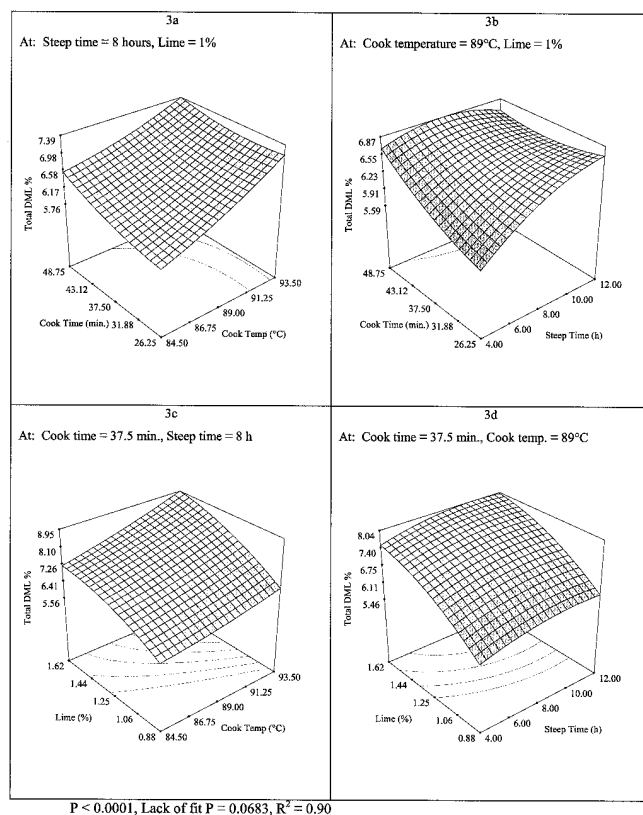


Fig. 3. Response surface graphs for total dry matter loss in nejayote and washwater.

being lost in either steepwater or washwater, it is not surprising that a model representing total loss better fits our data. It was evident from the slopes of the response surfaces (Fig. 3) and  $P$  values of regression coefficients (Table III) that lime concentration ( $R^2 = 0.0001$ ) was the most critical factor influencing total DML during nixtamalization. Lime concentration had more effect on DML when nixtamalization was conducted at higher cook temperatures. From the RSM graph plotted between cook time and steep time (Fig. 3b), it was apparent that total DML gradually increased during the first 8 hr of steeping and then leveled off or slightly decreased.

Pflugfelder et al (1988) studied DML in commercial corn masa production and concluded that overnight steeping accounted for most of the corn DML during nixtamalization. They also observed that greater losses during cooking did not result in an overall DML increase. Unlike the observation of Pflugfelder et al (1988), this study suggests that cook temperature and lime concentration during nixtamalization governed total DML while extended steeping for up to 12 hr did not increase DML significantly. Overnight steeping is often followed in the industry for convenience. These observations suggest that such a practice does not adversely influence corn solid loss.

### Nejayote and Washwater pH

Nejayote and washwater pH exhibited significant ( $P < 0.0001$ , lack-of-fit  $P = 0.0643$ ,  $R^2 = 0.92$ ) quadratic regression models with processing variables indicating a nonlinear relationship (Fig. 4). A decrease in nejayote pH at a constant lime concentration of 1% (Fig. 4a and b) suggests corn calcium uptake during nixtamalization. It was interesting to observe that at relatively lower cook temperatures nejayote pH increased, suggesting a desorption of corn calcium with increasing cook temperatures. However, at high-cook temperatures, nejayote pH rapidly decreased after  $\approx 30$  min of cooking, suggesting increased corn calcium absorption rate.

Figure 5 shows the RSM graphs obtained for changes in washwater pH with processing variables. An increase in washwater pH suggests loosely attached nixtamal calcium that readily transfers to

the wash on equilibration. Besides lime concentration used during nixtamalization, cook temperature significantly influenced washwater pH (Fig 5a and c). Nixtamalization of corn at high temperatures probably formed a stronger corn-calcium bond so that little was transferred to the wash.

## CONCLUSIONS

In the ranges studied, corn DML varied at 3.17–9.82%. Most of the DML was in nejayote (4.12–6.55%), while DML in wash varied at 1.08–2.23%. Cook temperature and percentage of lime added during nixtamalization were the most critical factors influencing total DML. Total corn DML increased during steeping, however, most of the loss occurred during the first 6-8 hr of steeping.

Khan et al (1982) observed that corn solid losses increased with increasing cooking time and temperature. However, steeping accounted for much of the loss. Pflugfelder et al (1988) observed that solid losses were 8.5–12.5% when corn was steeped for 16–24 hr, but losses were only 4.4% when the corn was steeped for  $\approx 5$  hr. They concluded that total losses were not directly proportional to cooking losses but were greatly influenced by steeping.

It is evident from this RSM study that cooking accounted for most of the solid loss, while steeping accounted for a relatively smaller portion of the loss. DML did not linearly increase on steeping for 12 hr but leveled off after  $\approx 6$ –8 hr of steeping. Similar observations were made when other corn hybrids, including softer yellow corn hybrids, were investigated for DML during steeping (data not shown). Cooking of corn at high-temperature long-time with no or minimal steeping is common in commercial masa and instant masa flour production facilities to expedite nixtamalization. It appears from this RSM study that such a practice would result in a higher DML as compared with nixtamalization at lower temperature with an extended steep time.

Cooking and steeping conditions influence masa quality. However, grinding conditions also play a significant role. By varying the amount of water added during grinding and the fineness of the

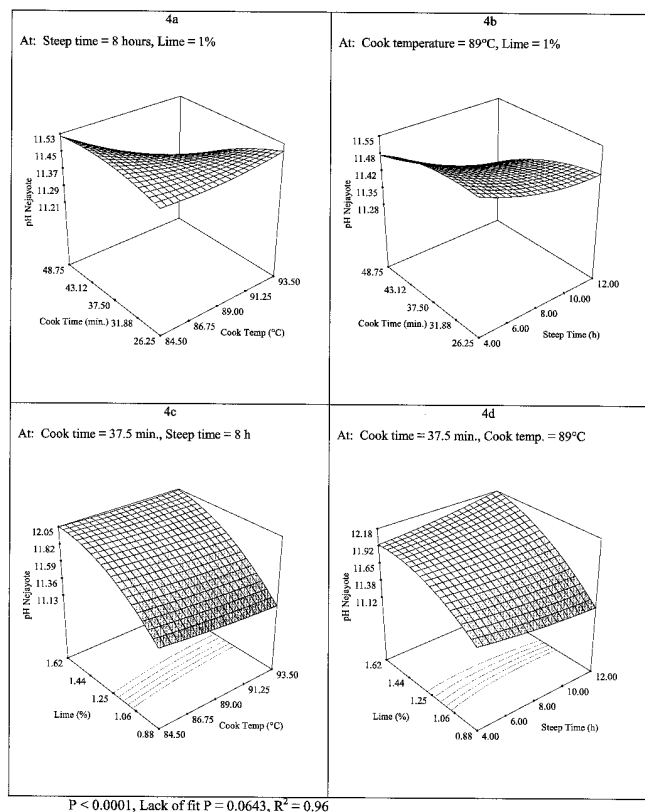


Fig. 4. Response surface graphs for nejayote pH.

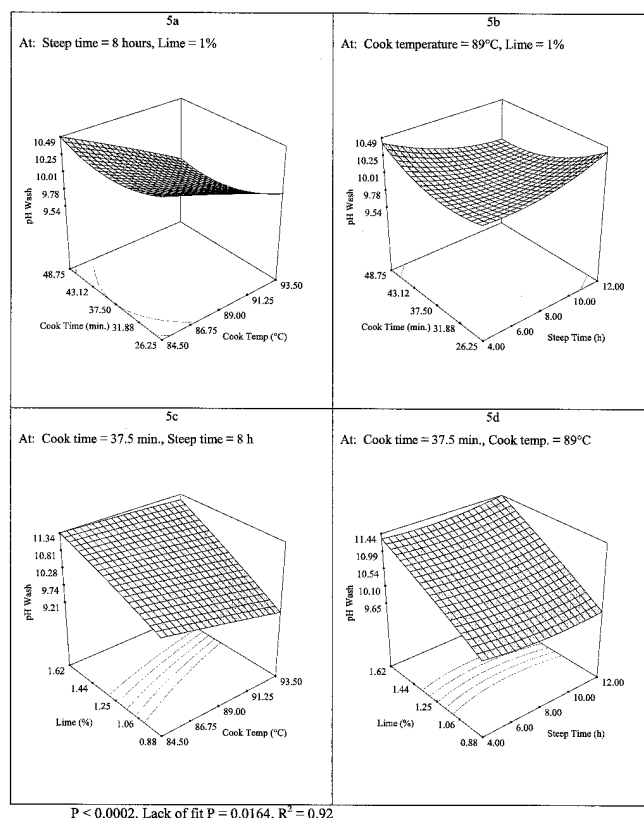


Fig. 5. Response surface graphs for washwater pH.

grind, masa texture can be significantly altered so that it becomes difficult to directly relate masa quality to nixtamalization conditions. When masa was produced from corn used in this study, we were able to suitably modify the grinding conditions so that we could produce tortillas from all the experimental trials (data not shown). Tortilla quality, however, was not directly related to total DML as high levels of pericarp removal did not improve tortilla quality. The overall best tortillas were produced when DML range was 6–6.2%; however, acceptable tortillas could be produced even when DML was <3.5%.

Nixtamalization cook-steep procedures in tortilla production facilities vary considerably. Most processors raise the temperature of water, corn, and lime mixture, and remove the heat supply during steeping. Others only add corn to a hot water-lime mixture and start steeping immediately, while large facilities may even try to cook rapidly at a high temperature and immediately wash and grind, eliminating or minimizing steeping. Such wide variation in nixtamalization protocols will certainly result in different DML results. However, all of these processes may end in producing masa of suitable quality by suitably altering the grinding conditions. It can be concluded that corn DML may be reduced and dry solid yield increased by following proper cooking and steeping protocols without adversely affecting masa quality. Further studies would be useful, however, to investigate the effect of cook-steep conditions on masa quality. For a given corn hybrid, it appears possible to optimize nixtamalization conditions to minimize effluent generation and corn solid loss without compromising masa quality.

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