Canola meal NIR calibration implementation
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Report prepared for the Co-operative Research Centre for High Integrity Australian Pork

By

Mr John Spragg
JCS Solutions Pty Ltd
32-34 Grantham Crescent
Berwick Vic 3806
Phone: 61 3 9769 7027
Fax: 61 3 9769 7174
Mobile: 0402 831 843
E-mail: jspragg1@optusnet.com.au

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Executive Summary

Previous project work has looked at the effects of heat processing on canola meal quality. Total and reactive lysine NIR calibrations have been developed for predicting variation in meal quality. The previous calibration statistical data identified the need for further samples to be analysed to improve the calibrations.

A total of 53 Canola meal samples were collected from eleven oilseed crushing plants. Additional purposely heat damaged samples were produced through autoclaving. Samples were analysed for total and reactive lysine, as well as wet chemistry parameters. The additional canola meal sample results were added to the NIR calibrations previously developed. New calibrations were developed using a FOSS 6500 NIR spectrophotometer.

The inclusion of more samples, as well as heat damaged autoclaved samples, has provided a greater spread of samples, with improved NIR calibration statistics. The RPD value for both total and reactive lysine has increased. The new calibrations have greater ability to distinguish between low, medium and high results.

This project work increased the number of samples the canola meal that total and reactive lysine NIR calibration equations are based on. This has provided an improvement in the calibrations statistics and the ability to predict meal quality.

The project has provided more robust NIR calibrations for prediction of total and reactive lysine content. The variability between commercial crushing plant samples identifies the value in utilising NIR calibrations in providing rapid and lower cost meal quality assessment. The revised calibration equations will be released for use by the oilseed, livestock and feed industries.

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1. Introduction

Previous project work jointly funded by Pork CRC and AOF (Spragg and Mailer 2007, 2008 and 2009, Spragg 2011) has looked at the effects of heat processing on canola meal quality. This work has generated total and reactive lysine NIR calibrations for use by industry in predicting variation in meal quality. Spragg (2011) in reporting total and reactive lysine calibration statistical data, identified the need for further samples to be analysed to further improve the calibrations.

This project will complete the canola meal NIR calibration work and result in the technology being delivered to industry.

The work involves analysis of additional samples, including intentionally heat damaged canola meal. An additional 60 samples, some of which have been autoclaved for different times, are being added to increase the range in values and to strengthen the NIR calibrations. The work will also provide additional reference samples for use during calibration transfer to end users.

2. Methodology

2.1. Sample Collection

A total of 53 Canola meal samples were collected from eleven oilseed crushing plants and sent to NSW I&I, Wagga Wagga Research Institute. Meal samples came from seven expeller (includes a cold press plant) and four solvent processing operations.

An additional seven samples were obtained from autoclaving canola meal for 0, 5, 10, 15, 20, 25 and 30 min at 135 °C. The autoclave was initiated with a 6.5 min steam charge to bring the chamber temperature and pressure up to 135°C and 217 Kpa, respectively. Then for the sterilising stage the chamber temperature and pressure remained as required (either 7, 14, 21 or 28 min and 217 Kpa). The sample was dried for 5 min at 110 °C and 14 Kpa before discharge from the chamber.

2.2. Sample Analyses

Canola meal samples were ground and analysed through an NIR spectrometer (FOSS 6500) generating spectral data at NSW I&I Wagga Wagga Research Institute.

Total and reactive lysine analysis was completed by DAFWA, utilising the methodology established by Massey University. Additional amino acids were analysed by HPLC.

Wet chemistry analysis including crude protein, crude fat, NDF, ADF, glucosinolates and sinapine was completed by NSW I&I Wagga Wagga Research Institute.

2.3. Calibration Development

The additional 60 canola meal sample results were added to the NIR calibrations previously developed. New calibrations were developed following the instrument manufacturers' directions using a FOSS 6500 NIR spectrophotometer.

3. Outcomes

3.1. Autoclave Results

Total and reactive lysine results for canola meal subject to autoclaving are shown in Table 1. Heat damage through autoclaving is shown in the reduction in total and reactive lysine. Figures 1 and 2 identify the greater

loss of reactive lysine with increasing heat processing, with this being a more sensitive measure of heat damage than total lysine content.

Table 1: Total and reactive lysine (g/kg) of canola meal autoclaved for varying time periods.

Time (Min)	Total Lysine	Reactive Lysine
0	17.5	16.6
5	16.2	15.4
10	15.6	13.8
15	14.9	12.0
20	13.1	9.2
25	11.0	7.8
30	11.6	8.0

Figure 1. Reactive/Total Lysine - canola meal autoclaved for varying time periods

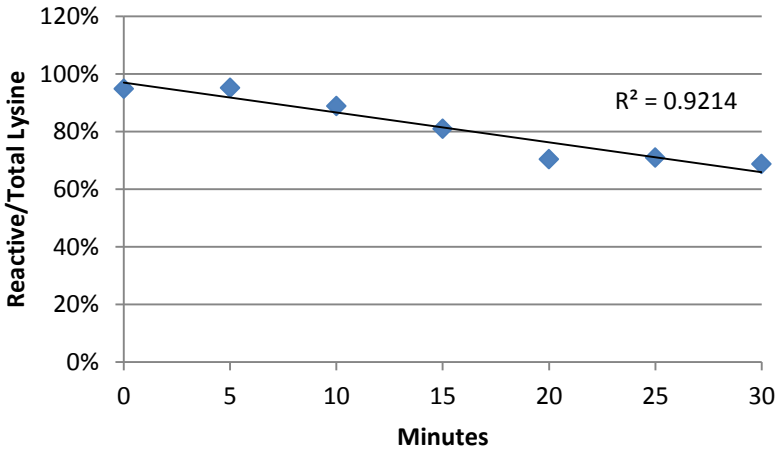
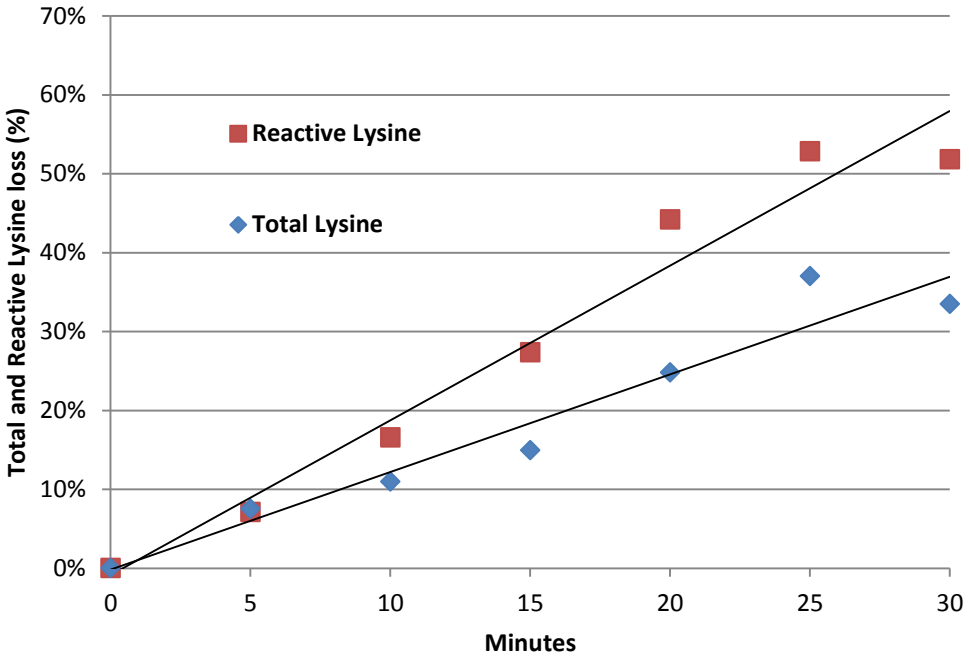


Figure 2: Total and reactive lysine loss - canola meal autoclaved for varying time periods



The autoclaved samples provided more significantly heat damaged samples to improve the NIR calibration robustness.

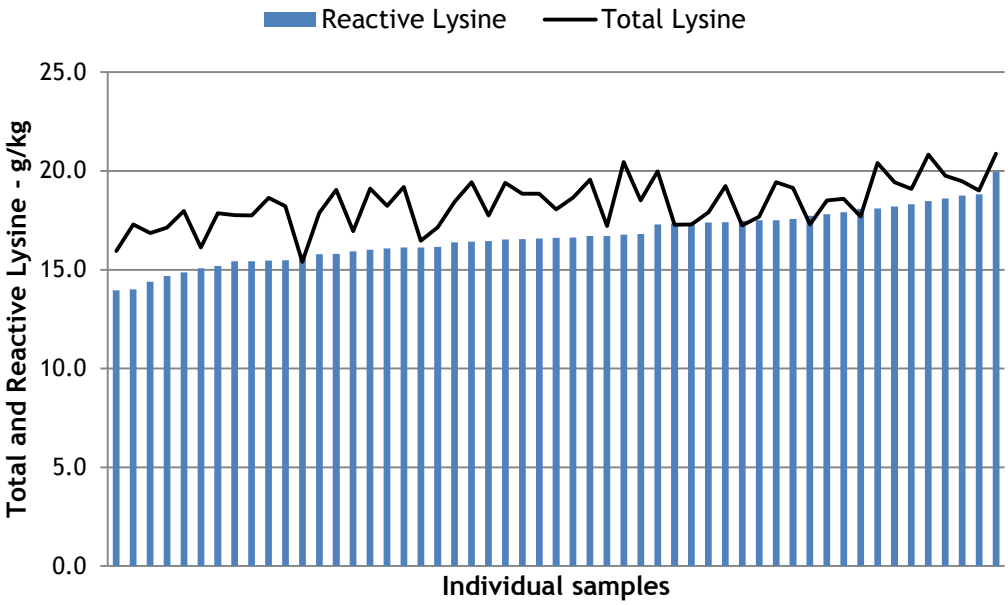
3.2. Canola Meal Analysis

Total lysine, reactive lysine and the ratio of reactive/total lysine results for the additional samples tested under this project work are shown in Table 2. Reactive lysine content ranged from 14.0 to 19.9 g/kg and was more variable than total lysine (Figure 3).

Table 2: Total lysine, reactive lysine and reactive/total lysine content of canola meal (g/kg, as received), min and max values are not from the same samples.

		Total Lysine	Reactive Lysine	Reactive/Total Lysine
Expeller	**Min**	15.4	14.0	85.0%
n = 30	**Mean**	17.9	16.8	93.8%
	Max	19.7	18.8	102.5%
	Std Dev	1.09	1.30	
Solvent	**Min**	16.1	14.0	81.0%
N = 23	**Mean**	18.9	16.6	87.6%
	Max	20.9	19.9	96.4%
	Std Dev	1.14	1.36	

Figure 3: Variation in reactive lysine content over total lysine



Wet chemistry and amino acid results are shown in tables 3 and 4.

Table 3: ***NDF, ADF, crude protein, crude fat, glucosinolates and sinapine content of canola meal (as received)***

	Nutrient	NDF	ADF	Crude Protein	Crude Fat	Glucosinolates	Sinapine
	Units	%	%	%	%	umoles/g	g/kg
Expeller	Min	22.0	15.0	30.2	8.4	3.0	9.0
n = 30	Mean	26.3	18.8	34.5	10.3	8.7	10.3
	Max	33.0	25.0	37.2	16.8	12.0	12.0
	SD	2.80	2.30	1.78	2.12	2.07	0.84
Solvent	Min	24.0	16.0	32.5	0.4	2.0	6.0
N = 23	Mean	25.8	18.3	37.3	1.7	3.5	8.2
	Max	28.0	20.0	41.1	3.4	8.0	10.0
	SD	1.30	1.21	2.02	0.72	2.16	0.83

Table 4: Amino acid content of canola meal (g/100g, as received)

Amino Acid	Histidine	Glycine	Threonine	Cysteine	Alanine	Arginine	Tyrosine	Valine	Methionine	Phenyl alanine	Isoleucine	Leucine	Lysine
Min	0.62	1.33	1.30	0.74	1.20	1.67	0.92	1.39	0.64	1.07	1.05	1.91	1.54
Mean	0.85	1.74	1.61	1.01	1.53	2.10	1.15	1.74	0.83	1.38	1.37	2.42	1.83
Max	1.04	2.09	1.97	1.20	1.86	2.60	1.35	2.17	1.07	1.87	1.81	3.01	2.09
SD	0.09	0.13	0.11	0.11	0.11	0.16	0.09	0.13	0.09	0.14	0.12	0.19	0.12

n = 53

3.3. NIR Calibrations

Calibration statistics are provided in Table 5 for total and reactive lysine. Shown are the previous calibration equation statistics and the changes with inclusion of the additional samples from this project work.

The inclusion of more samples as well as heat damaged autoclave samples has provided a greater spread of samples, with improved calibration statistics. The RPD value for both total and reactive lysine has increased, this identify the calibrations as having greater ability to distinguish between low, medium and high results.

Table 5: NIR calibration statistics for reactive lysine and total lysine in canola meal

Description	N	Mean	SD	Min	Max	RSQ	SECV	1-VR	RPD
Previous Stage 3 calibrations									
Total lysine (g/kg, as received)	126	19.425	1.633	15.821	24.440	0.901	0.764	0.780	2.14
Reactive lysine (g/kg, as received)	124	16.612	1.851	11.773	23.521	0.843	0.927	0.750	2.00
Revised Stage 4 calibrations									
Total lysine (g/kg, as received)	184	18.974	1.905	13.258	24.690	0.901	0.766	0.838	2.49
Reactive lysine (g/kg, as received)	183	16.454	2.001	10.447	22.480	0.863	0.934	0.782	2.15

SD, Standard deviation; RSQ, Square of correlation co-efficient R; SECV, Standard error or cross validation; 1-VR, Coefficient of determination for cross validation, RPD; Ratio of prediction to deviation (SD/SECV).

The relationship between the wet chemistry values and the NIR values for total lysine and reactive lysine are shown in Figures 4 and 5.

Figure 4: Relationship between wet chemistry and NIR for reactive lysine content (as received) in canola meal.

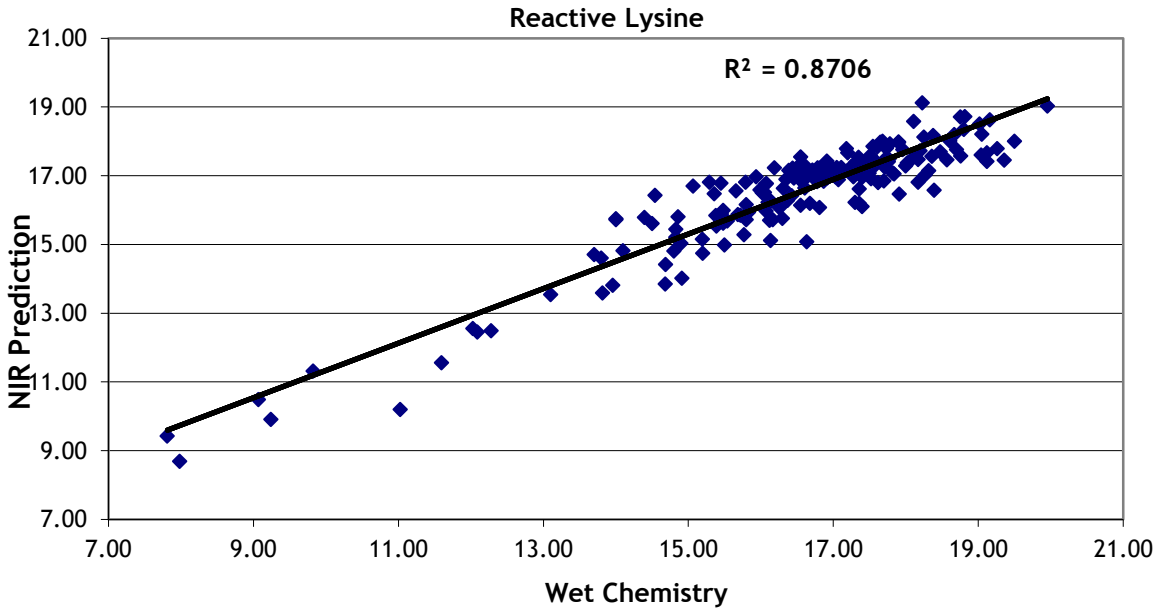
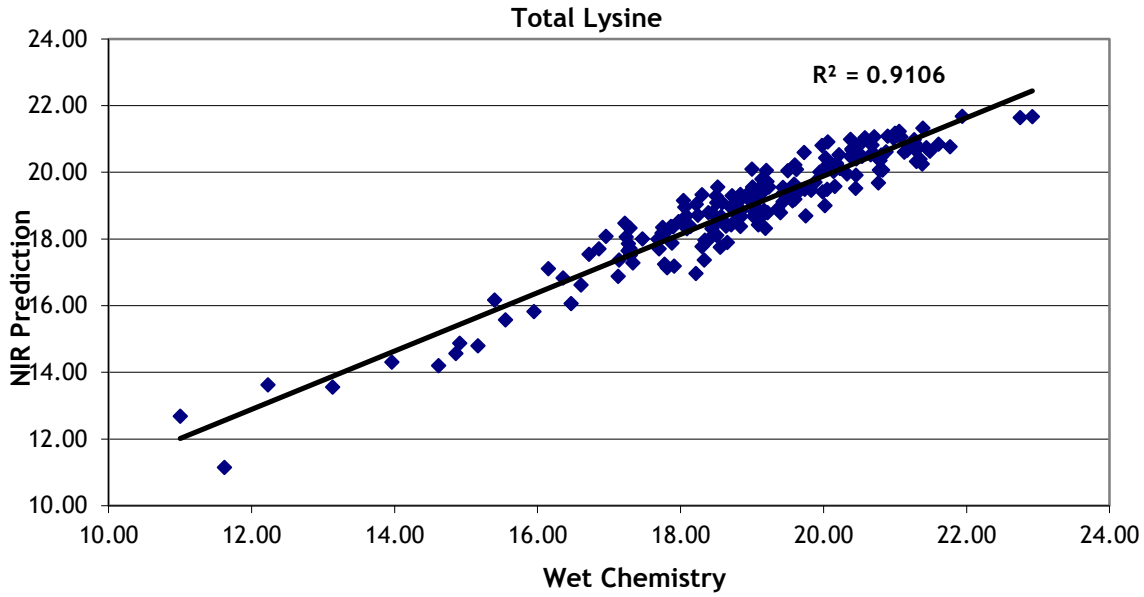


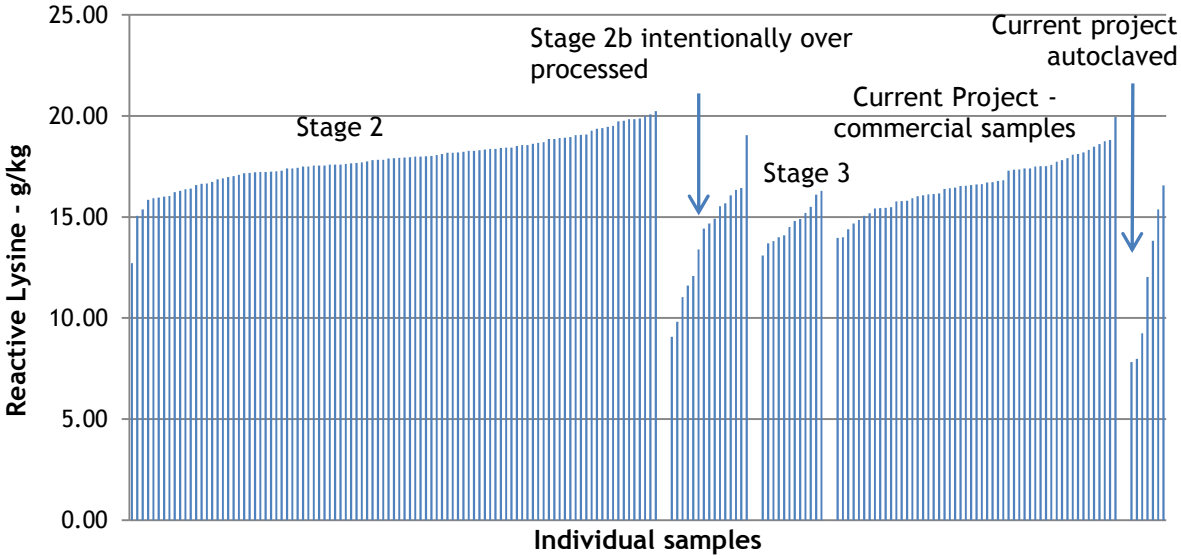
Figure 5: Relationship between wet chemistry and NIR for total lysine content (as received) in canola meal.



4. Application of Research

The use of autoclaving to generate a range of heat damaged samples has been previously used for soybean meal (Kim and Mullan 2012 and 2013) and canola meal (Almeida et al 2013). More extreme heat damaged samples were collected by Spragg and Mailer 2008 from crushing plants that were instructed to intentionally over process meal, this being achieved through longer retention times during processing. In this current project work, autoclaving generated samples with lower reactive lysine and lower reactive/total lysine ratios than seen in commercial meal. Figure 6 identifies the autoclaved sample reactive lysine results relative to other samples, including those intentionally over processed in Stage 2b.

Figure 6: Reactive lysine content of canola meal samples from previous and current project work (Spragg and Mailer 2008, 2009 and Spragg 2011).



Canola meal samples from commercial crushing plants have provided total and reactive lysine results consistent with previous data collection. Residual fat in canola meal is a function of the processing equipment and extraction methods is use by each of the eleven crushing plants. Figures 7 and 8 shows the range in protein and fat content of cold press, expeller and solvent extraction canola meal plants. Both cold press and expeller plants have higher residual fat content and generally lower protein than meal from solvent processing plants. There is however variation between plants with this also being influenced by the canola seed stock being processed and whether oil refining takes place on site with gums being added back to the finished meal.

Figure 7:Canola meal crude protein content(as received)

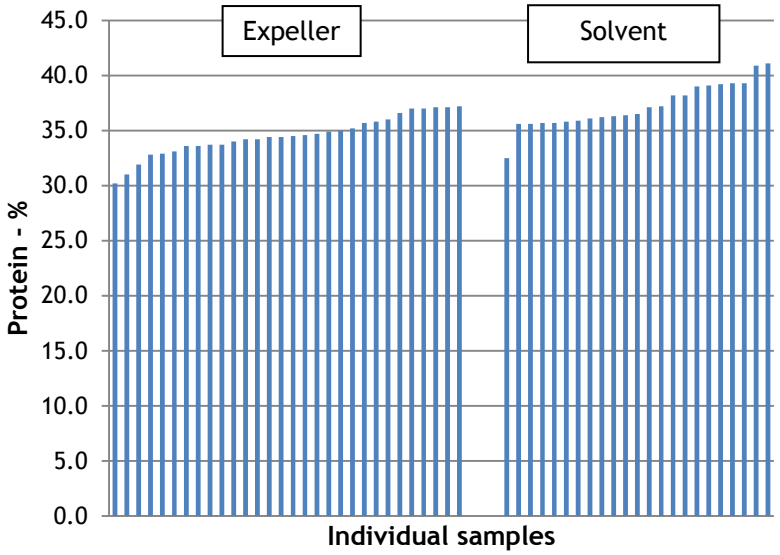
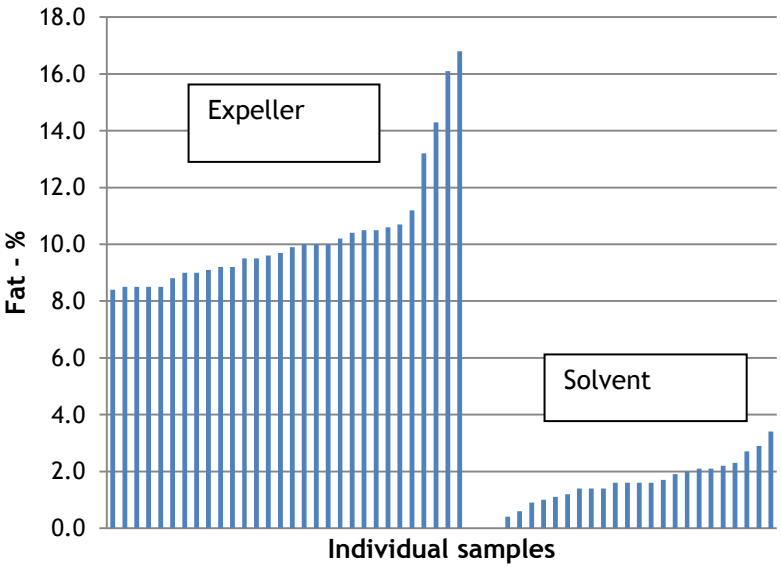


Figure 8: Canola meal crude fat content (as received)

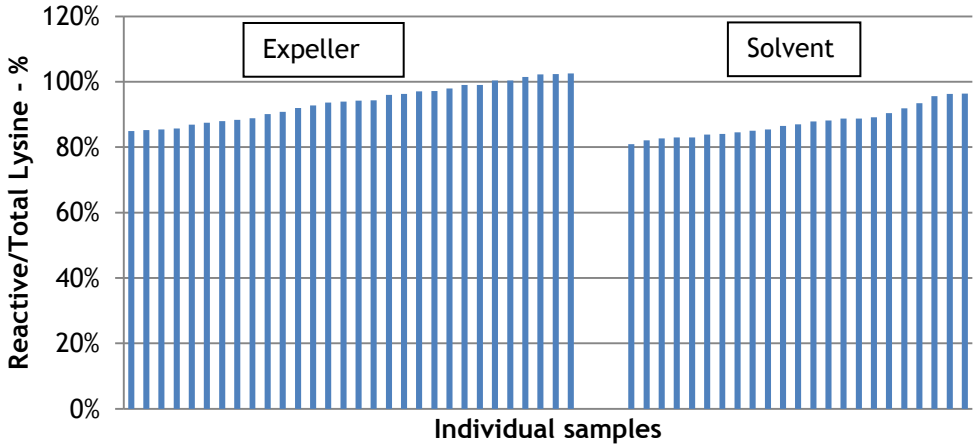


Reactive lysine has been shown to provide a measure of the available lysine in heat processed feedstuffs (Rutherford 1997). Work by Kim and Mullan (2013) using soybean meal has identified a direct relationship between reactive lysine and pig ileal digestible bioavailable lysine content. The variation in reactive/total lysine in canola meal samples supplied for oilseed crushing plants is shown in Figure 9.

For the samples tested, both expeller and solvent meal samples show high variation in lysine bioavailability ranging from 80 to 100%. Lower reactive lysine and protein damage was identified as occurring with higher levels of heat processing (Spragg and Mail, 2007). The data from this project identifies that heat damage occurs in both expeller and solvent processing and crushing plants can influence meal quality for monogastric feeding through the plant operation.

When looking at individual crushing plant results, there was a trend for plants to produce consistent meal quality results as determined by reactive/total lysine. i.e. either consistent high >95%, medium 85-95% or low <85%. Due to commercial sensitivity, individual crushing plant datum is not available for release. While having a high lysine bioavailability is considered favourable for pig and poultry feeding, the ruminant industries recognise benefits in higher heat processing resulting in slower rumen degradability.

Figure 9: Canola meal reactive lysine content



5. Recommendations

This project work has increased the number of samples the canola meal total and reactive lysine NIR calibration equations are based on. This has provided an improvement in the calibrations statistics and the ability to predict meal quality.

The project has provided more robust NIR calibrations for prediction of total and reactive lysine content. The variability between commercial crushing plant samples identifies the value in utilising NIR calibrations in providing rapid and lower cost meal quality assessment.

It is recommended that the revised calibration equations be released for use by the oilseed, livestock and feed industries.

6. References

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