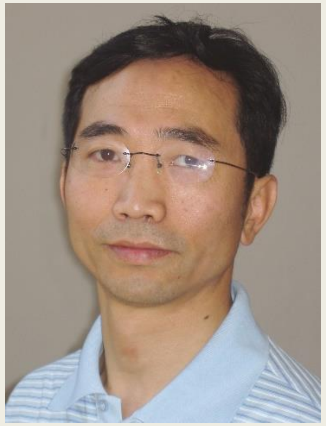

Roger Ruan

Professor Ruan is the Director of Center for Biorefining and Professor of Bioproducts and Biosystems Engineering Department and Food Science and Nutrition Department at University of Minnesota, and Fellow of ASABE. He has published over 400 papers in refereed journals, books, and book chapters, and over 300 meeting papers and other reports, and holds 18 US patents. He is also a top cited author in the area of agricultural and biological sciences. He has supervised over 65 graduate students, 110 post-doctors, research fellows, and other engineers and scientists, and 12 of his Ph.D. students and 8 other post-doctors hold university faculty positions. He has received over 170 projects totaling over $40 million in various funding for research, including major funding from USDA, DOE, DOT, DOD, and industries like ABC. He has served as guest editor and/or editorial board member of Bioresource Technology, etc. and Editor-in-Chief and chairman of the board for International Journal of Agricultural and Biological Engineering.

NON-THERMAL PROCESSES FOR MAKING CLEAN LABEL FOOD AND NUTRACEUTICAL INGREDIENTS FROM ALMOND HULL

Roger Ruan
and
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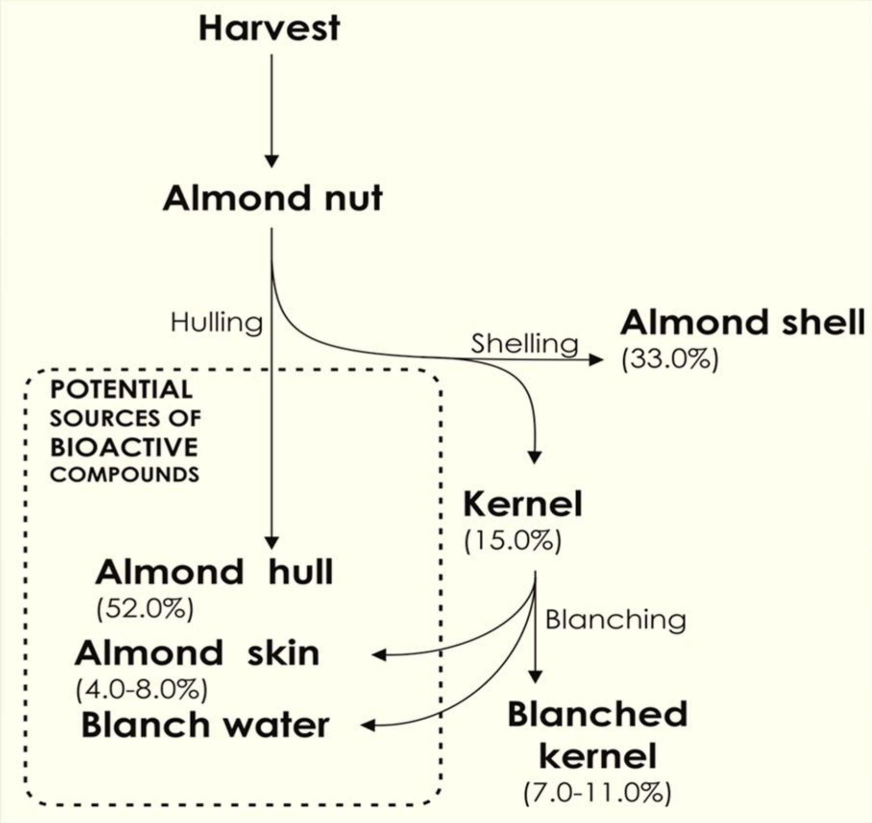
Department of Bioproducts and Biosystems Engineering
Department of Food Science and Nutrition
Center for Biorefining
University of Minnesota

california almonds™
Almond Board of California

Project background

- California almond industry generates a large amount of almond hulls (For every 3 pounds of almond kernels produced, 5 pounds of hulls are generated. It produced 3.37 billion pounds of hulls in 2016)
- Almond hulls are traditionally sold as a feed supplement for California dairy cows
- Almond hulls contain high level of antioxidants. Almond extracts were found to slow down oxidative processes in food products. Medical studies found that phytochemicals in almonds inhibited DNA niching and human LDL cholesterol oxidation
- The crude fibers of almond hulls have the potential to be converted to functional dietary fibers and other high value products



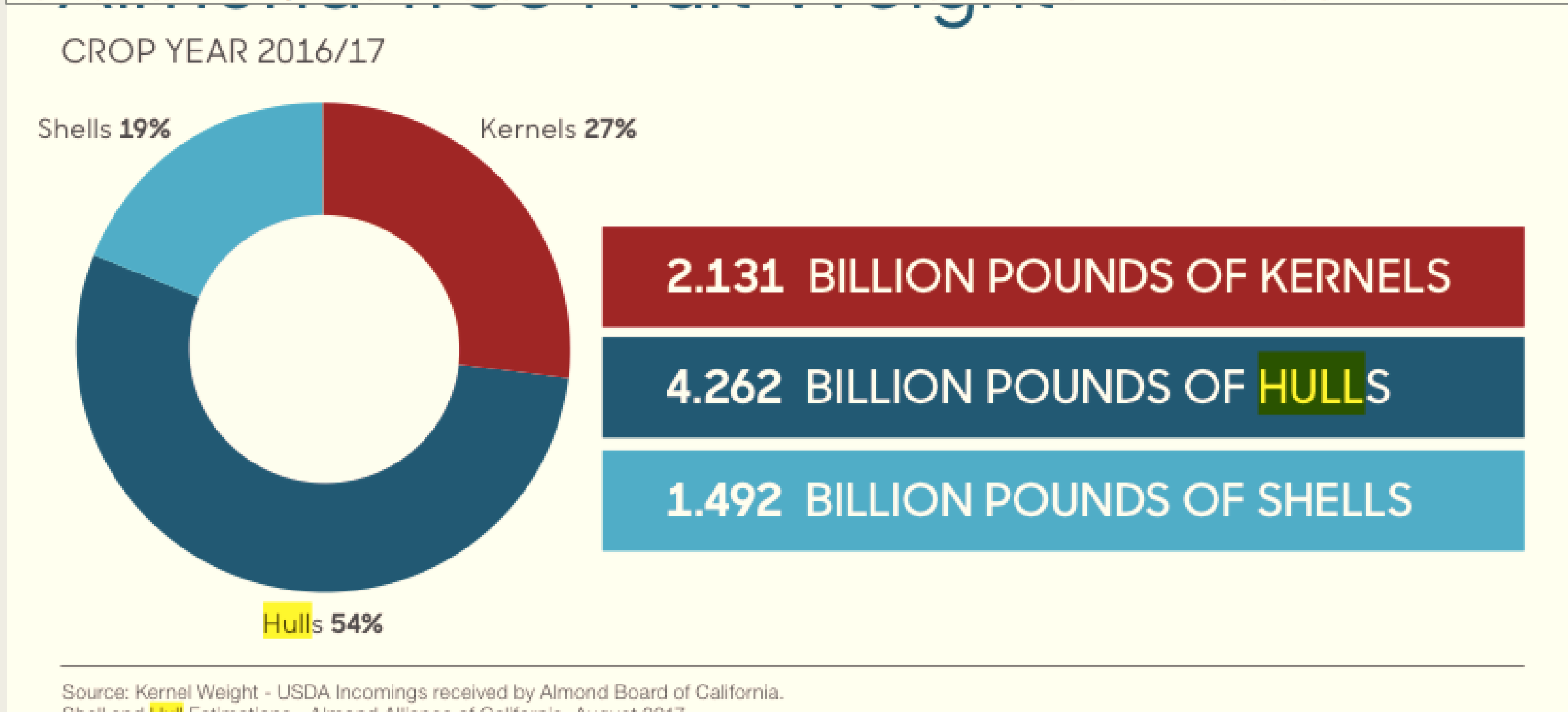
General Almond Lifecycle

Current market

- Major use as animal feed – ‘FEED into FOOD’
- The low milk price and bounded taxes impact the price of almond hulls, which incites the expanded market and other usage of almond hulls
- Soil amendments
- Plastic additives + colorant
- Anaerobic soil disinfestation

CURRENT MARKET

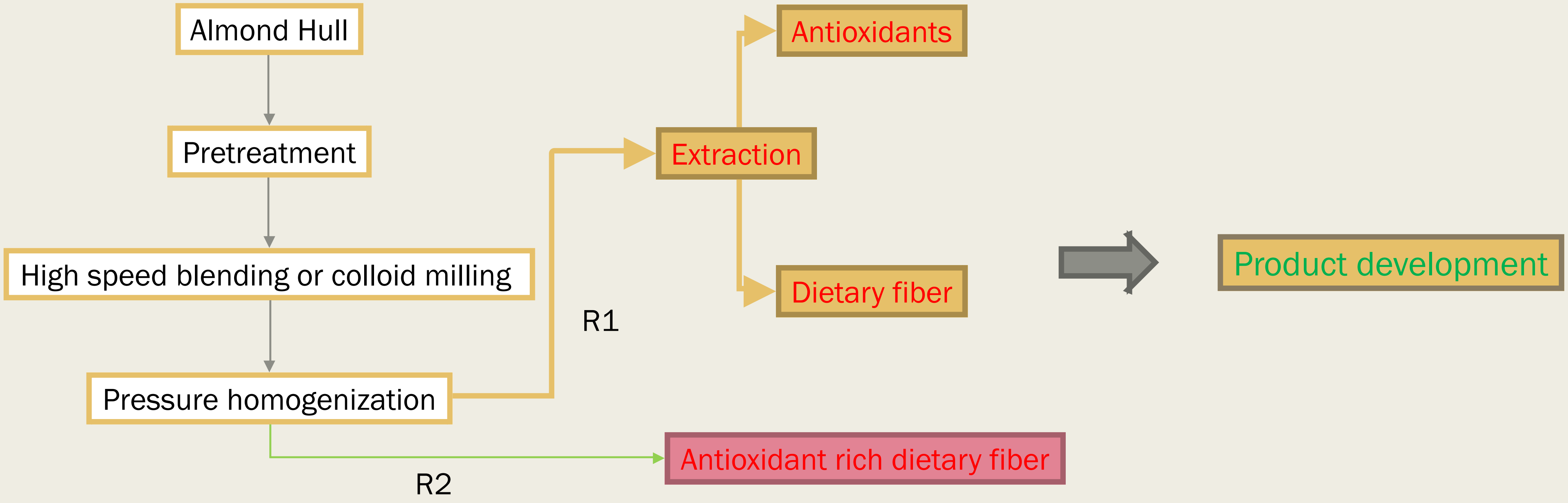
Distillers Dry Grains			
Rail to California Points		219.00	up 15.00-8.00
FOB Truck to California Points		205.00-223.00	dn 5.00-unch
Corn Gluten Pellets, 21 pct			
FOB California Points		175.00-183.00	up 5.00-1.00
Canola Meal			
FOB Central San Joaquin Valley		340.00-343.00	up 40.00-33.00
Rail Central San Joaquin Valley		NA	
Whole Almond Hulls			
FOB Huller Butte/Colusa County	Ltd	85.00	unch
FOB Huller Madera County	Ltd	105.00	unch
FOB Huller Fresno County	Ltd	105.00	unch
FOB Huller Kern County	Ltd	110.00	unch
FOB Huller Modesto/Oakdale/Turlock/Merced	Ltd	100.00	unch
FOB Huller Stanislaus County		NA	
Delivered Stockton/Modesto/Oakdale/Turlock		95.00-110.00	dn 5.00-unch
Delivered Hanford/Tulare/Visalia		105.00-115.00	dn 5.00-unch
Delivered Los Angeles		114.00-130.00	unch
Delivered Kern/Bakersfield	Ltd	120.00	unch



Project background

- The **goal** of the proposed research is to develop and evaluate processes to convert almond hull to antioxidant and dietary fiber rich ingredients for food and nutraceutical applications. The successful outcome of the research will lead to practical utilization of almond hull for production of value-added products, which can potentially bring sizable extra income to the almond producers and processors.

Approach


Almond Hull
Pretreatment
High speed blending or colloid milling
Pressure homogenization
R1
Extraction
Antioxidants
Dietary fiber
Product development
R2
Antioxidant rich dietary fiber

PREVIOUS RELATED WORK

Fiberstar Technology

- A motivating transition from patented technologies to commercialization

Improve Whole Grain Bioavailability and Sensory Quality

- A USDA funded major project

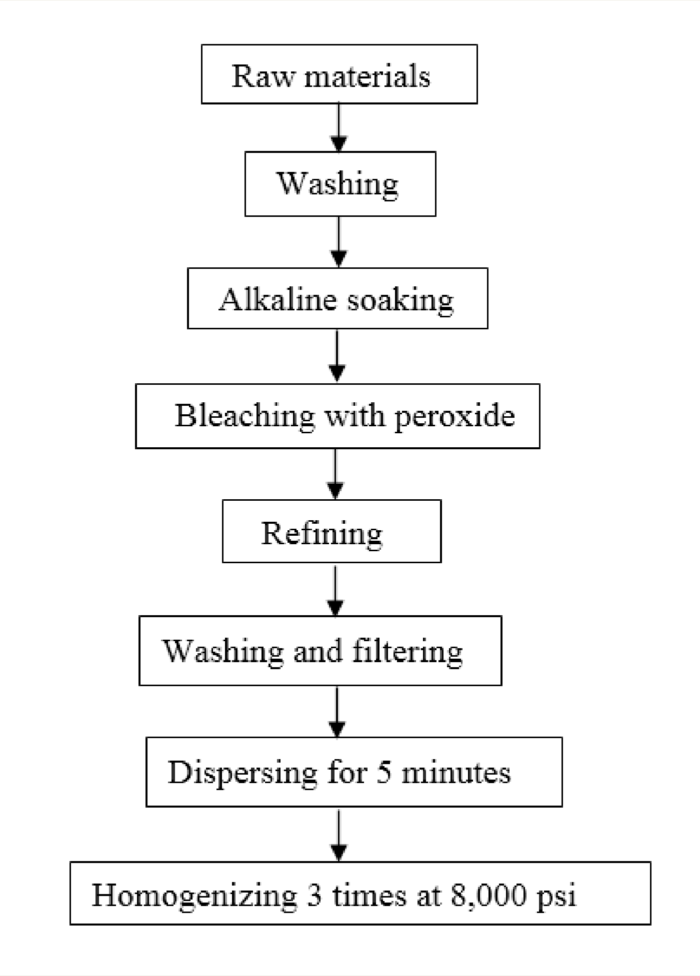
Publications

- Xing, J.-j., Cheng, Y., Shan, L., Chen, P., Lia, D., Ruan, R. 2017. Effect of High-Pressure Homogenization on the Extraction of Sulforaphane from Broccoli (Brassica oleracea) Seeds. Journal of Agricultural and Food Chemistry, Submitted.
- Zhu, X., Cheng, Y., Chen, P., Peng, P., Liu, S., Li, D., Ruan, R. 2016. Effect of alkaline and high-pressure homogenization on the extraction of phenolic acids from potato peels. Innovative Food Science & Emerging Technologies, 37, 91-97.
- Chen, P., Yanling, C., Shaobo, D., Xiangyang, L., Guangwei, H., Ruan, R. 2010. Utilization of almond residues. International Journal of Agricultural and Biological Engineering, 3(4), 1-18.
- Liu, C., G. Fu, Z. Tu, H. You, P.L. Chen, R. Ruan. 2004. Functional polysaccharides from lilies. *Transactions of ASAE,* 48(1):257-261*.*
- Lin, G., R. Ruan, P. Chen, W. Wilcke, and P. Addis. 2001. Structure-function relationships of highly refined cellulose. *Transactions of ASAE.* 44(6):1707-1712*.*

US patents

- Ruan, R., L. Gu, B. Lundberg, L. Chen, J. Johnson, and P. Addis. 2009. Cellulose fiber-based compositions and their method of manufacture. US Patent No: 7,582,213. Issue date: September 1, 2009.
- Ruan, R., B. Lundberg, L. Gu, L. Chen, J. Johnson, and P. Addis. 2006. Cellulose fiber compositions and films and their method of manufacture. US Patent No: 7,074,300. Issue date: July 11, 2006.
- Ruan, R., L. Xu, L. Yi, L. Chen, J. Johnson, and P. Addis. 2000. Cellulose fiber compositions and films. U.S. Patent No.: 6,083,582. Issue date: July 4, 2000.
- Chen, L., Ruan, R., L. Xu, L. Yi, J. Johnson, and P. Addis. 1998. Cellulose fiber compositions and film and the process for their manufacture. U.S. Patent No.: 5,817,381. October 6, 1998.
- Ruan, R., L. Xu, and P. Chen. 1998. Water vapor permeability and tensile strength of cellulose based composite edible films. *Applied Engineering in Agriculture* 14(4): 411-413.
- Ruan, R., J. Han, P. Chen, and B. Martinez. 1997. Study of temperature sensitive hydrogel using pulse NMR. *Biotechnology Techniques* 11(4): 257-260.
- Ruan, R., Y. Lun, J. Zhang, P. Addis, and P. Chen. 1996. Structure-function relationships of highly refined cellulose made from agricultural fibrous residues. *Applied Engineering in Agriculture* 12(4): 465-468.

Materials and process



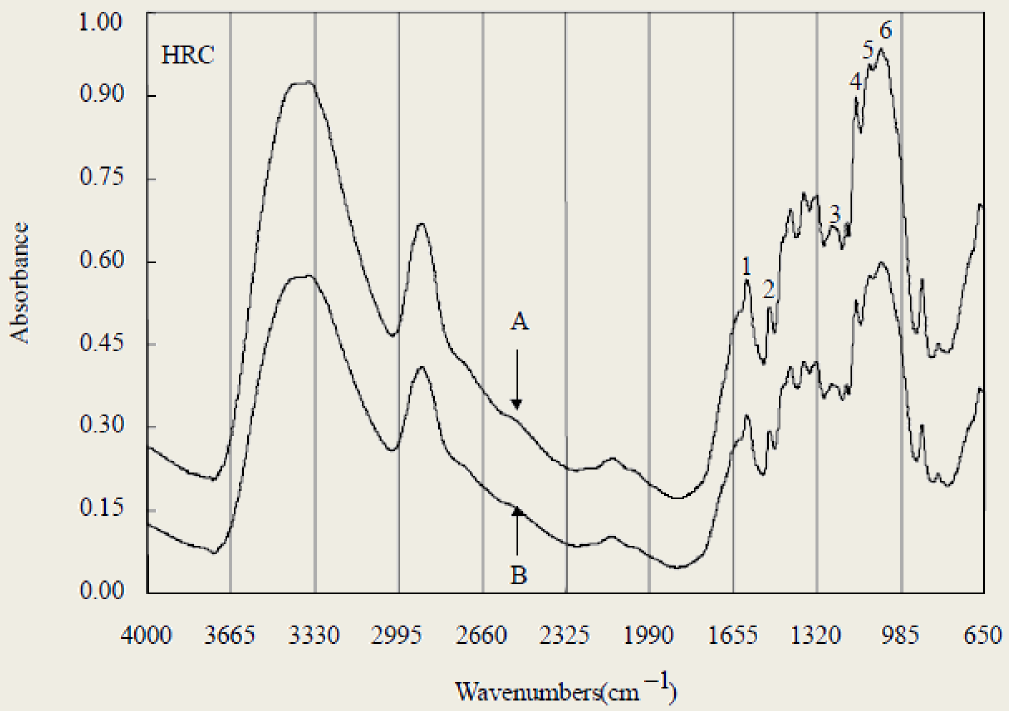
- Raw materials
  - *Corn stovers*
  - *Corn cobs*
  - *Soy hulls*
  - *Sugar beet pulp*
  - *Citrus pulp*
  - *Citrus peels*
  - *Wheat brans*
  - …

Process

- Raw materials
- Chopping or Grinding
- Alkaline digestion
- Refining
- Pulverization
- HRC

Size reducing

Chemical structure of HRC not altered



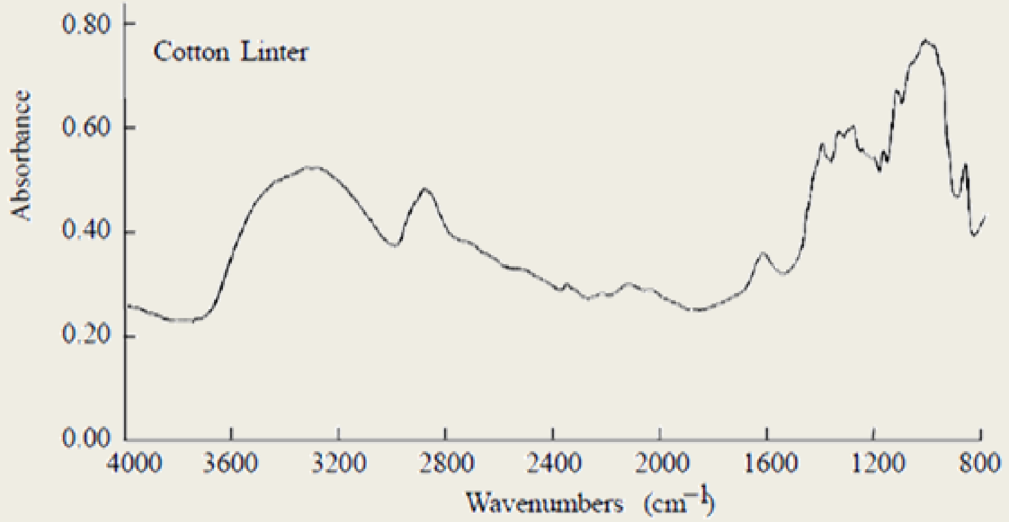


Figure 1. DRIFTS spectra of HRC materials: A = HRC sample with crystallinity ratio 2.0, B = HRC sample with crystallinity ratio 1.7, and cotton linter (Schultz and McGinnis, 1985). Infrared bands of interest: 1 = 1600 cm^{-1}, 2 = 1510 cm^{-1}, 3 = 1425 cm^{-1}, 4 = 1170 cm^{-1}, 5 = 1100 cm^{-1}, and 6 = 1070 cm^{-1}.

Physiochemical properties

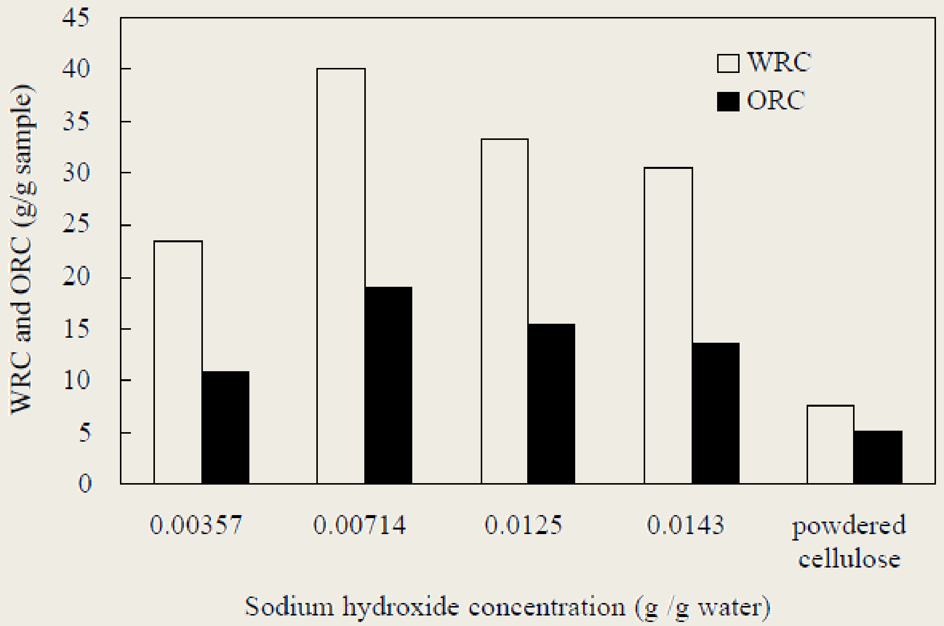


Figure 2. WRC and ORC of HRC and powdered cellulose (powdered cellulose data from Ang, 1991).

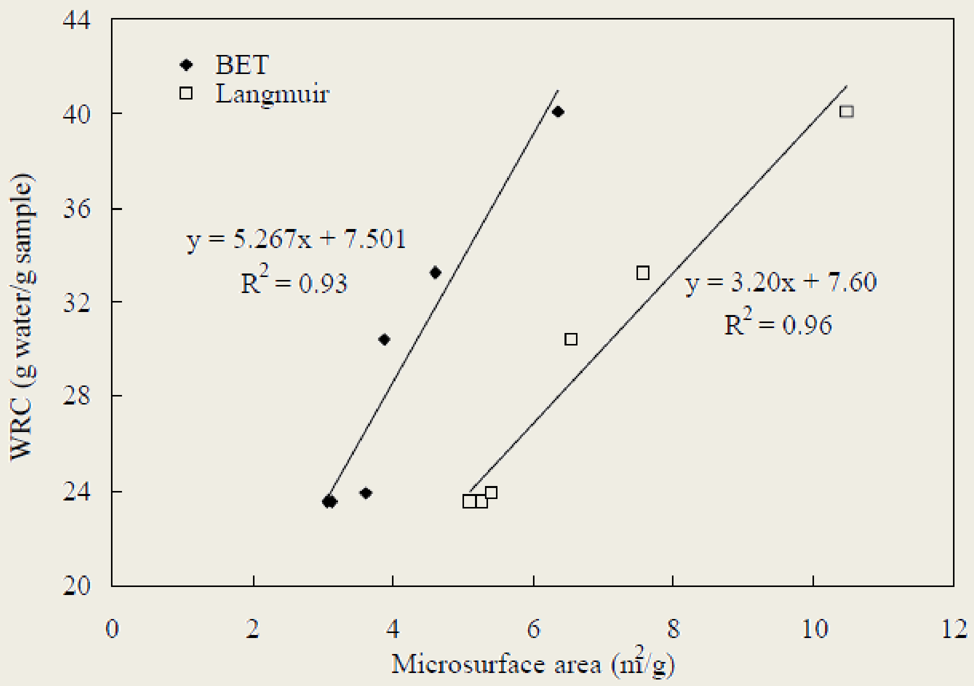
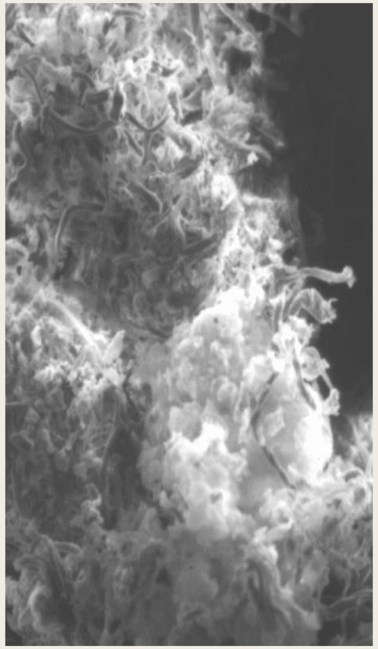
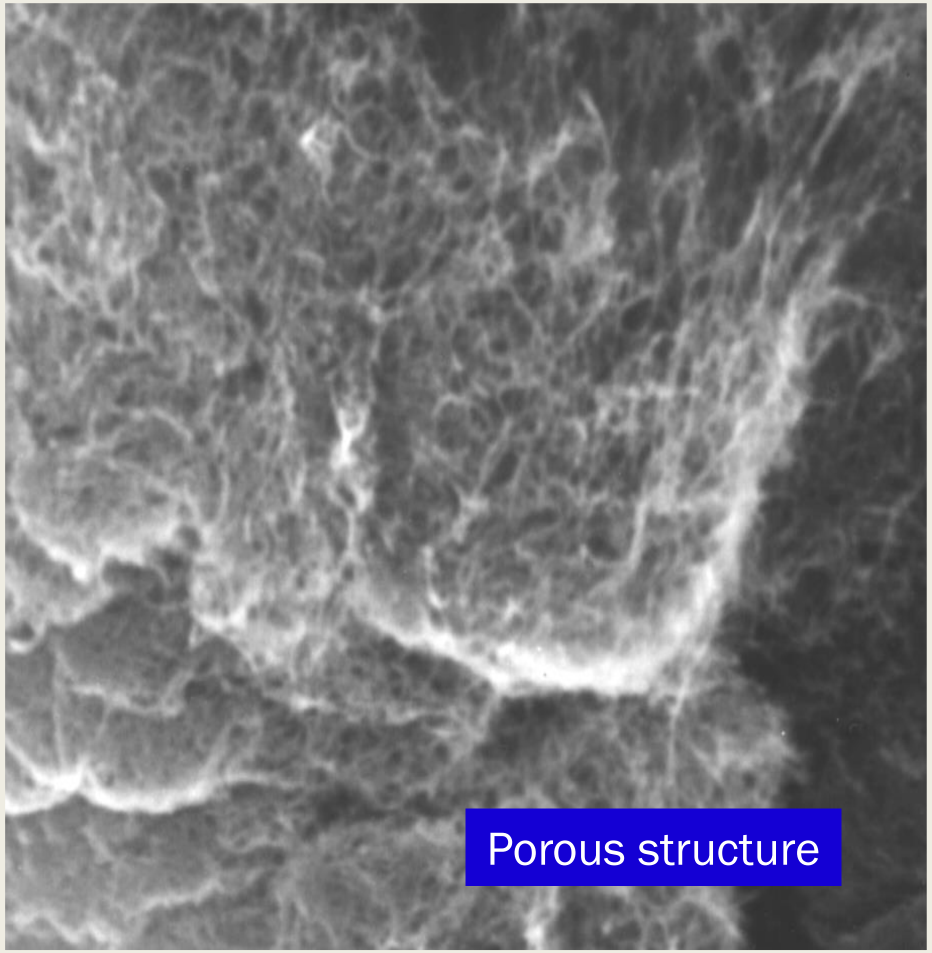


Figure 3. Microsurface area and WRC of HRC.



Cellulose fiber
(x100)

HRC
(x100)



Scanning electronic microscopic pictures

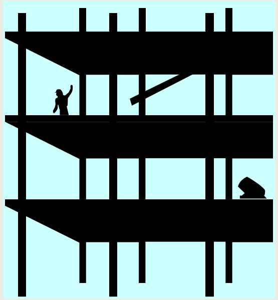
Properties and Applications of HRC

- High water holding capacity
- High viscosity
- Large surface area
- Smooth texture
- Zero calorie

- Dietary fibers
- Emulsion stabilizer
- Thickener
- Moisture control
- Foam stabilizer
- Ice crystal control
- Suspending agent
- Edible films and coatings

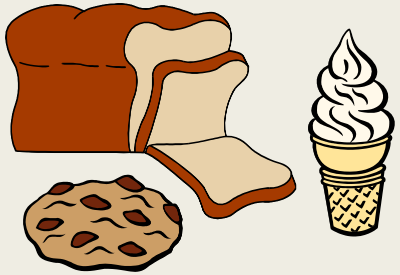
Filter pad

Construction materials



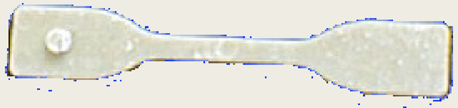
HRC aqueous gel

Powdered HRC



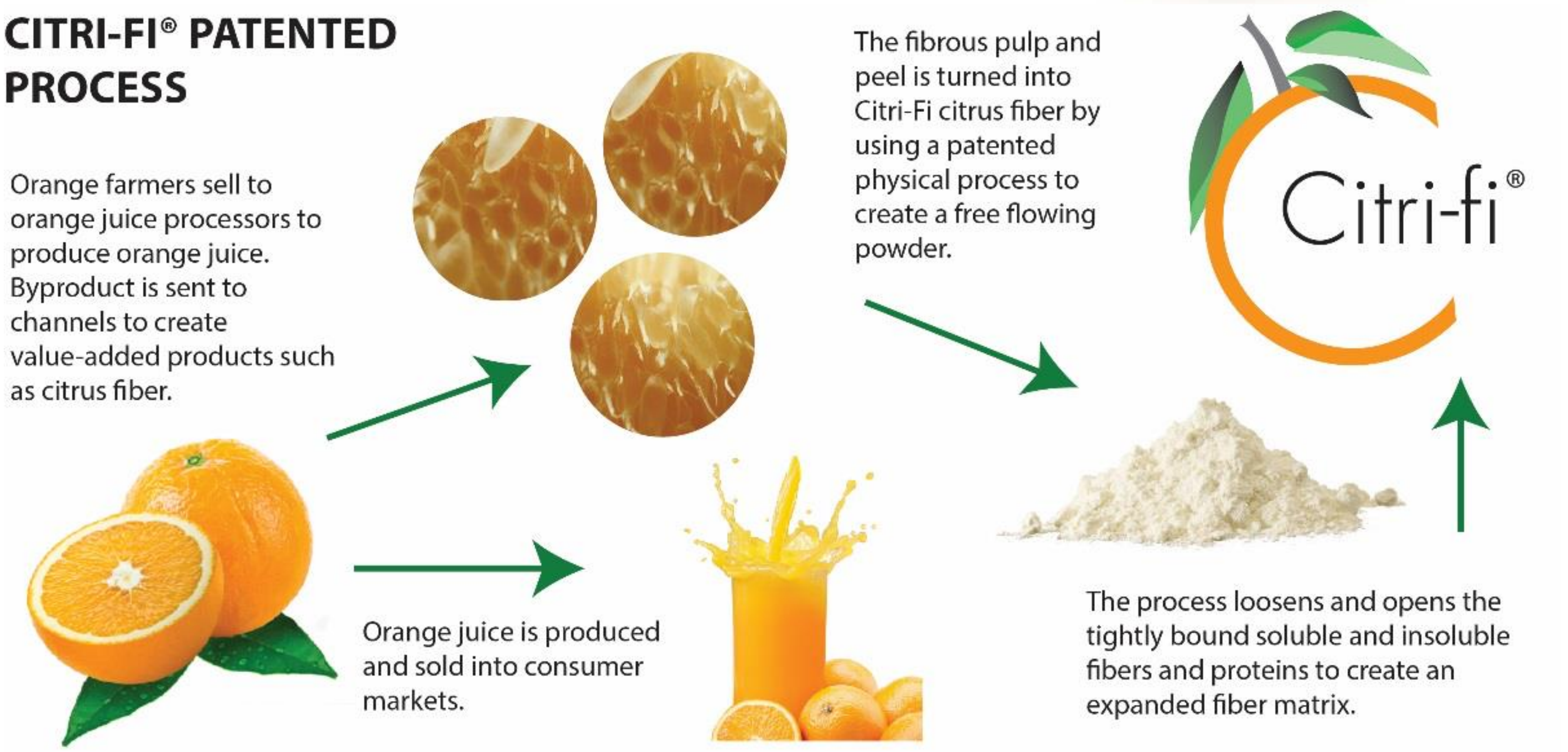
Fat replacer

Dietary fiber supplement



Press molded particles

Citri-Fi Manufacturing Process



Bakery Benefits

- Regular & Gluten-free Breads
  - Moisture retention
  - Improved quality over time
- Muffins & Cakes
  - Moisture retention
  - Reduced oil or egg
- Bakery Fruit Fillings
  - Pectin extension
  - Reduced blow-outs

Meat & Poultry Benefits

- Injection/Marinades
  - Phosphate replacement
  - Yield improvement
  - Reduced purge
- Ground Meats
  - Firm texture
  - Juicy texture
- Vegetarian Meats
  - Firm & juicy texture
  - Binding




Dressing & Sauces Benefits

- Tomato-based Sauces
  - Tomato extension
  - Reduced syneresis
- Dressings
  - Replace/reduce egg
  - Emulsification
- Salsas & Dips
  - Reduced syneresis
  - Improved texture

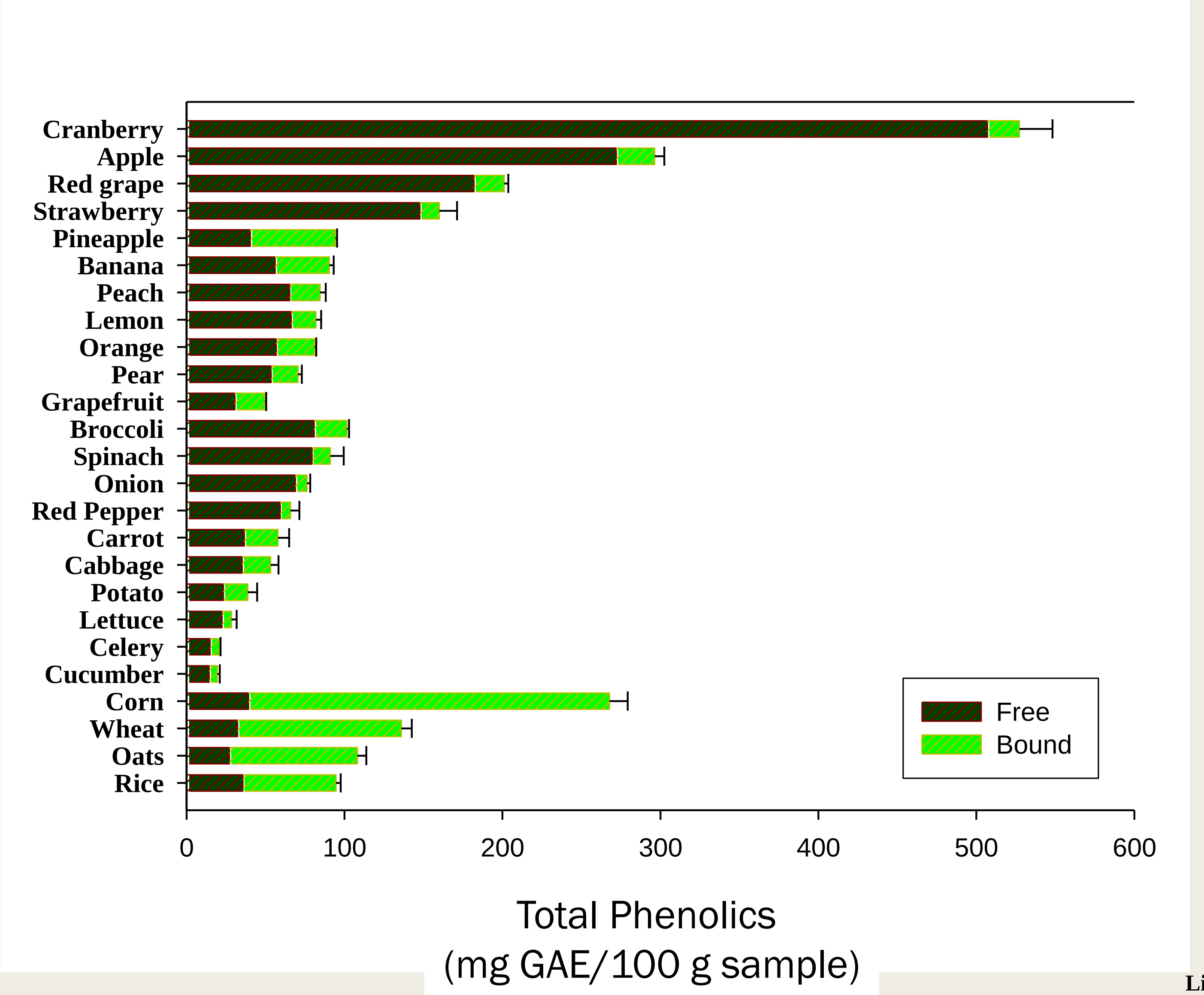
Dairy Benefits

- Yogurts
  - Reduced syneresis
  - Pectin extension in fruit preparation
- Ice Cream
  - Reduced ice crystallization
  - Replaces synthetic stabilizers
- Creams
  - Reduced syneresis
  - Dairy extension

Improve Whole Grain Bioavailability and Sensory Quality - Anti-oxidants and functional dietary fibers from wheat bran

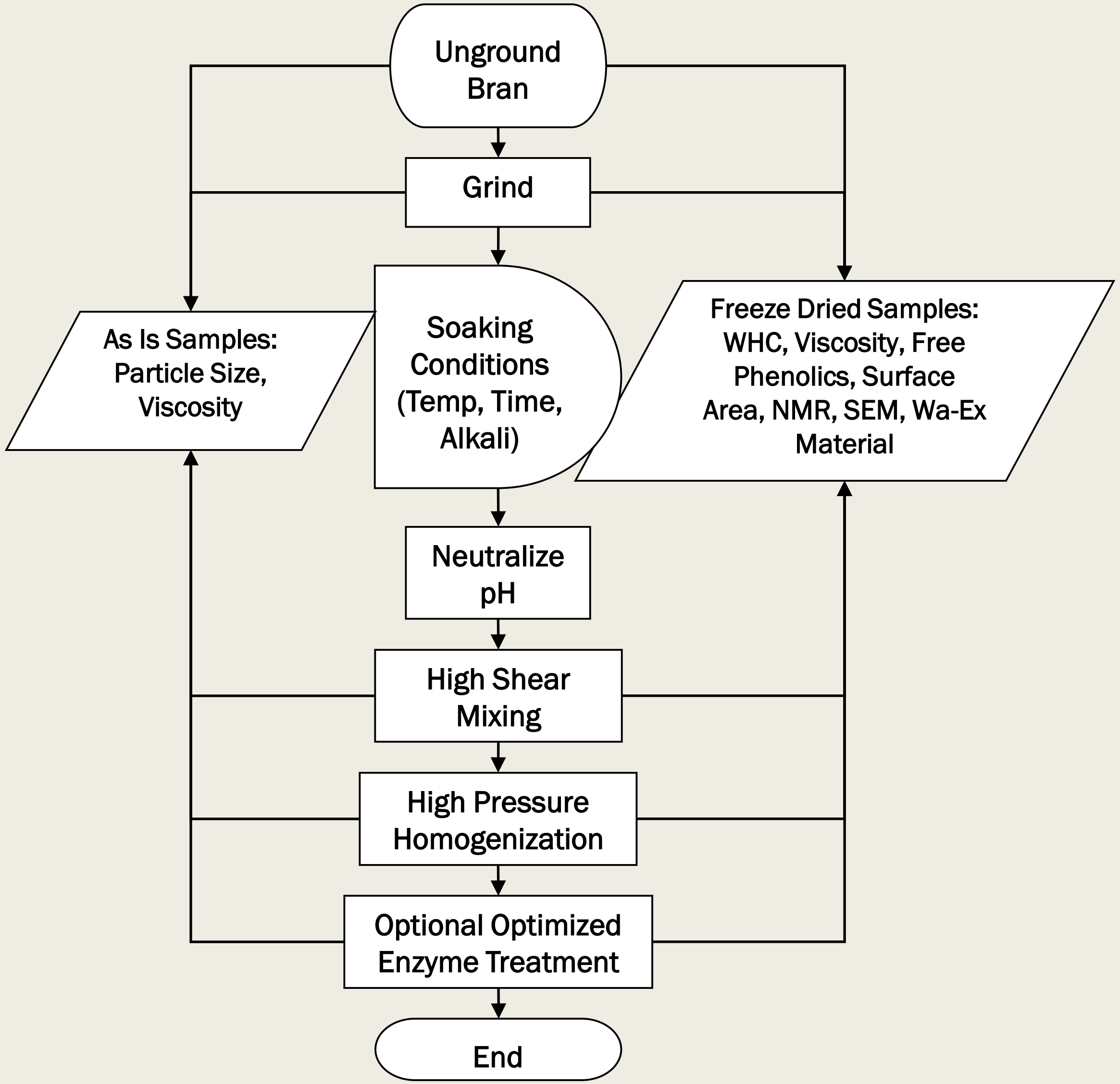
- Grain bran including aleurone layers is a major source of bioactive compounds such as dietary fiber, antioxidants, vitamins, minerals (potassium and magnesium), and phytochemicals (phytic acid and phenolic acids).
- These bioactive compounds supposedly provide a major portion of the health benefits associated with whole grain intake.
- However, as in other foods of plant-origin, the bioaccessibility and bioavaialability of these nutrients and phytochemicals in grain bran are limited by the cellular structure matrix.
- In addition to its limited bioavailability of phytochemicals, bran is also a major contributor to the undesirable sensory attributes of whole grain products.
- Hypothesis: Processing would improve the functionality and bioavailability of phytochemicals in cereal brans, which in turn will yield health benefits of cholesterol lowering and reducing oxidative stress and at the same time improve the sensory quality of whole grain products

Bound and free phenolics in plant materials

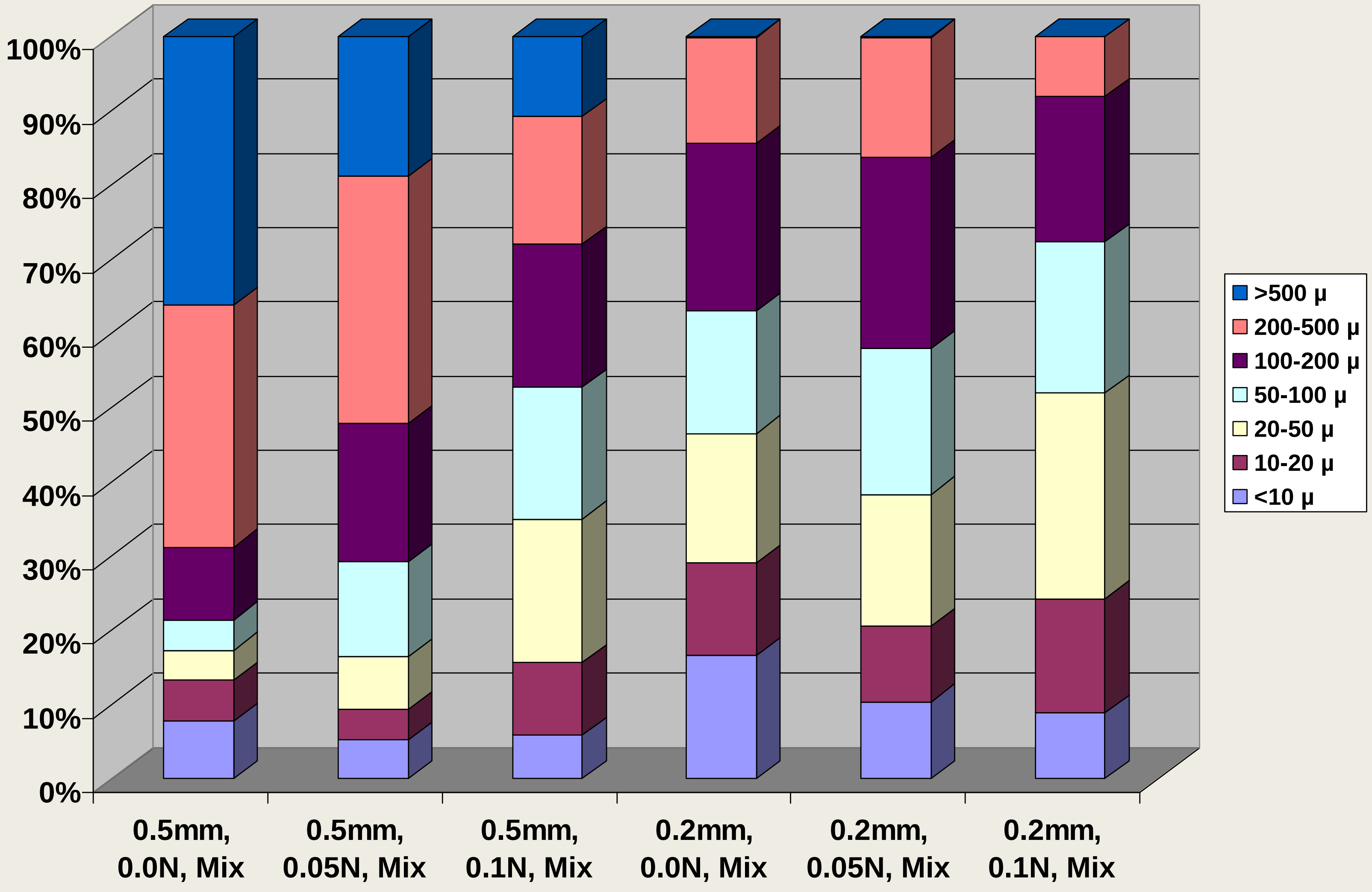


Liu, R.H. Journal of Cereal Science 46 (3): 207-219, 2007
(adapted from Sun et al, 2002, Chu et al, 2002, Adom and Liu, 2002)

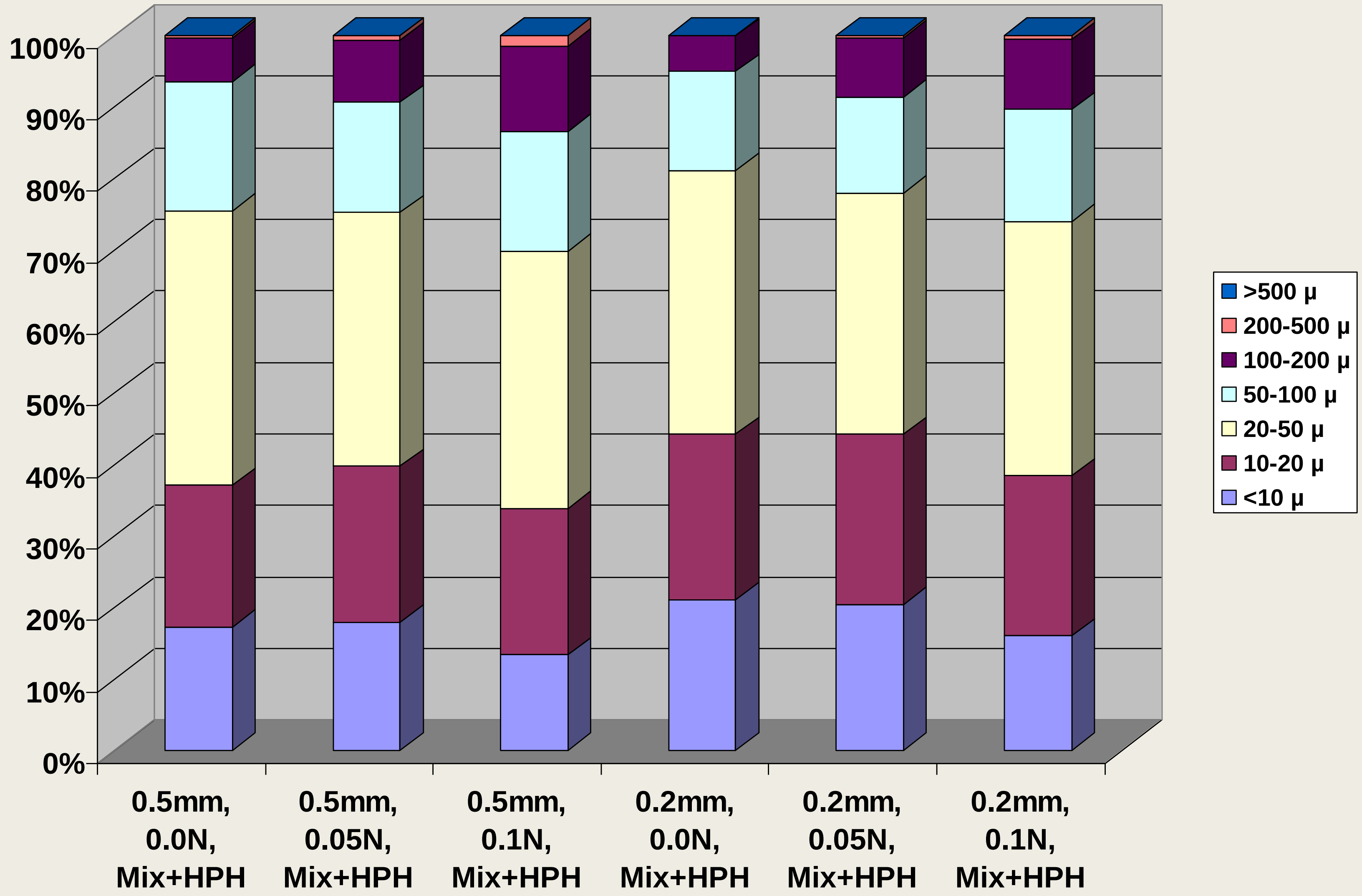
HPH Bran Processing Flowchart
Shows Process Steps, Collection Points, and Analyses.



% Particle Size Distribution of Processed Wheat Bran: High Shear Mixing Only

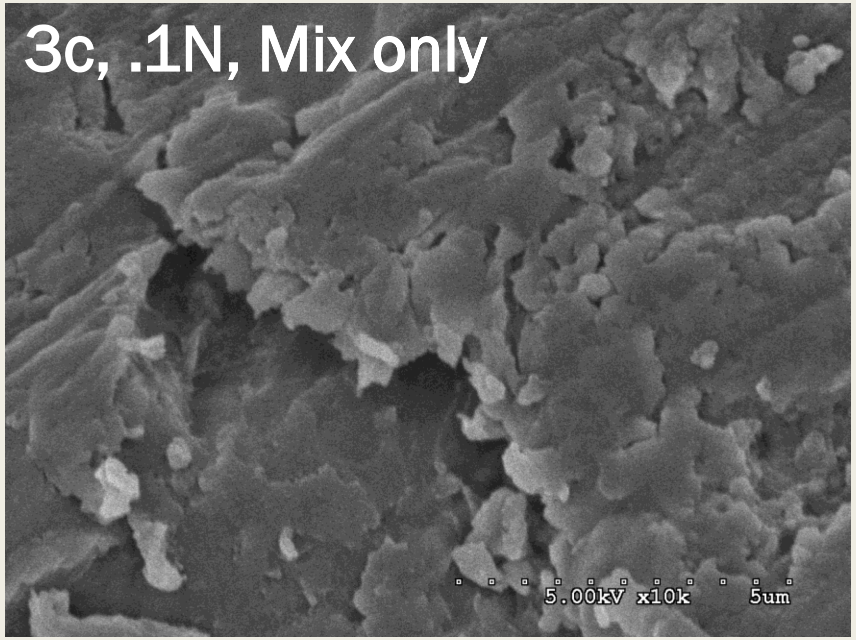


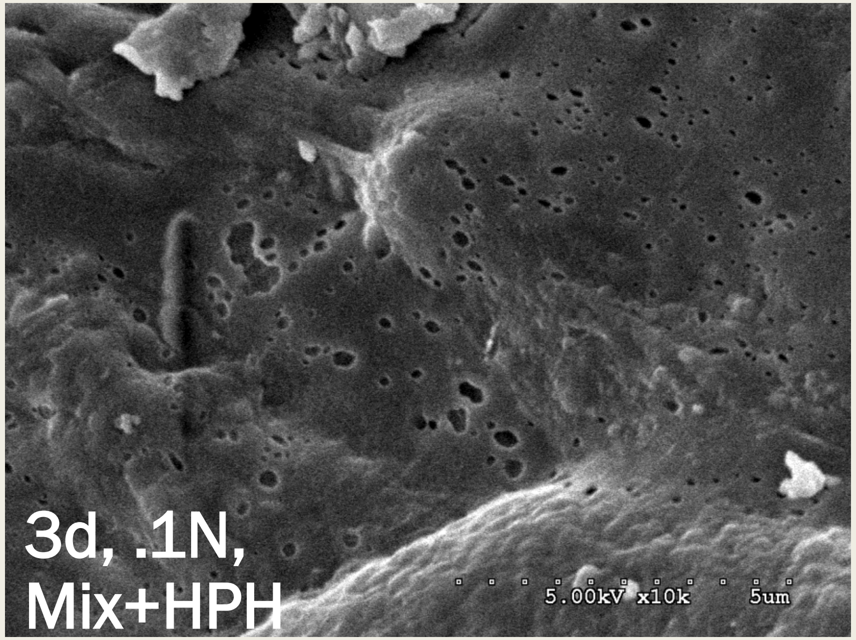
% Particle Size Distribution of Processed Wheat Bran: High Shear Mixing + High Pressure Homogenization

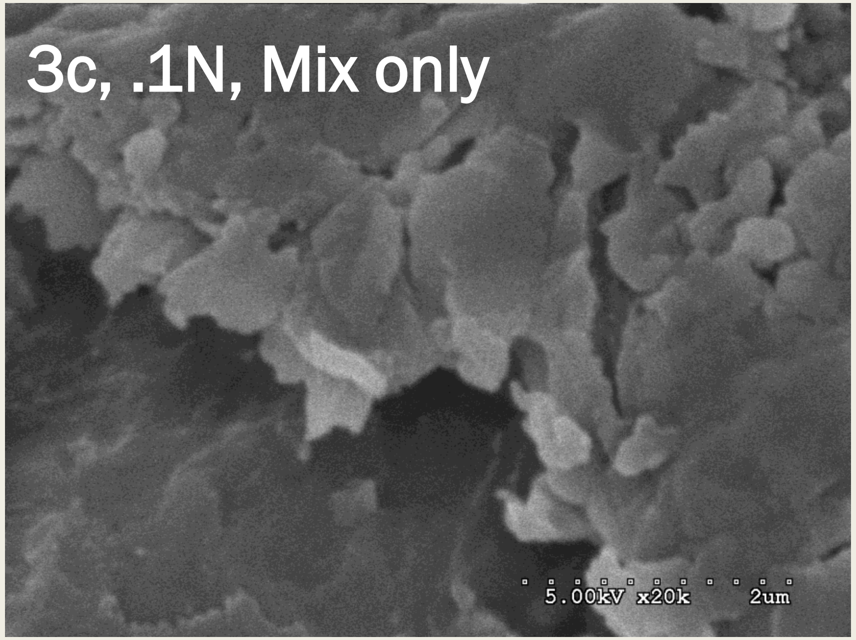


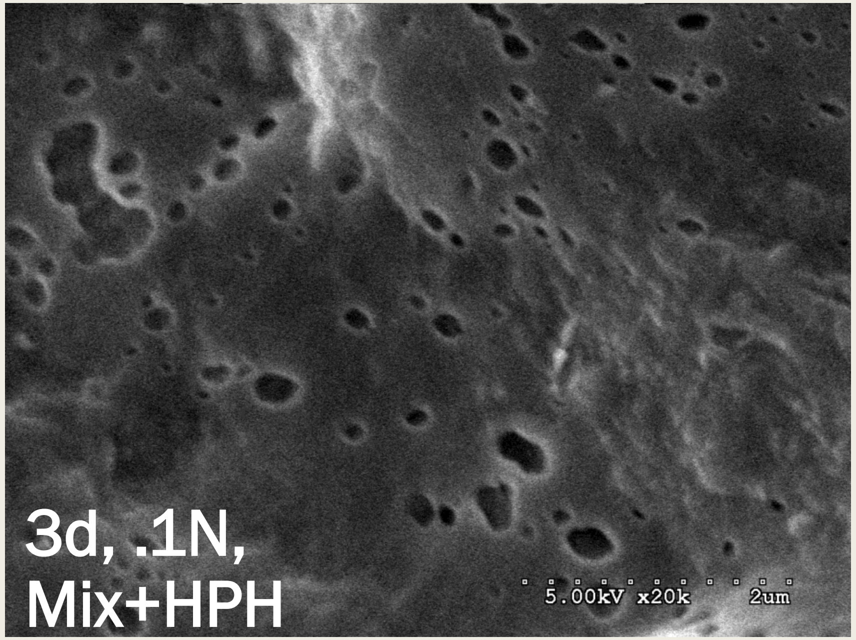
Viscosity increase, principally due to NaOH treatment, but starting bran size also significant

Bran Grind (through Retsch Mill screen)	**0.5mm**	**0.5mm**	**0.5mm**	**0.2mm**	**0.2mm**	**0.2mm**
NaOH Concentration (24 hour shaking at 60˚ C)	**0.0N**	**0.05N**	**0.1N**	**0.0N**	**0.05N**	**0.1N**
High Shear Mixing (24K rpm, 5 min)	**Mix**	**Mix**	**Mix**	**Mix**	**Mix**	**Mix**
% Viscosity Increase (vs 21132 control, 2% soln.)	**0%**	**122%**	**212%**	**27%**	**71%**	**114%**
High Pressure Homogenization (2 passes, 23K psi)	**Mix+HPH**	**Mix+HPH**	**Mix+HPH**	**Mix+HPH**	**Mix+HPH**	**Mix+HPH**
% Viscosity Increase (vs 21132 control, 2% soln.)	**75%**	**169%**	**187%**	**-1%**	**59%**	**158%**

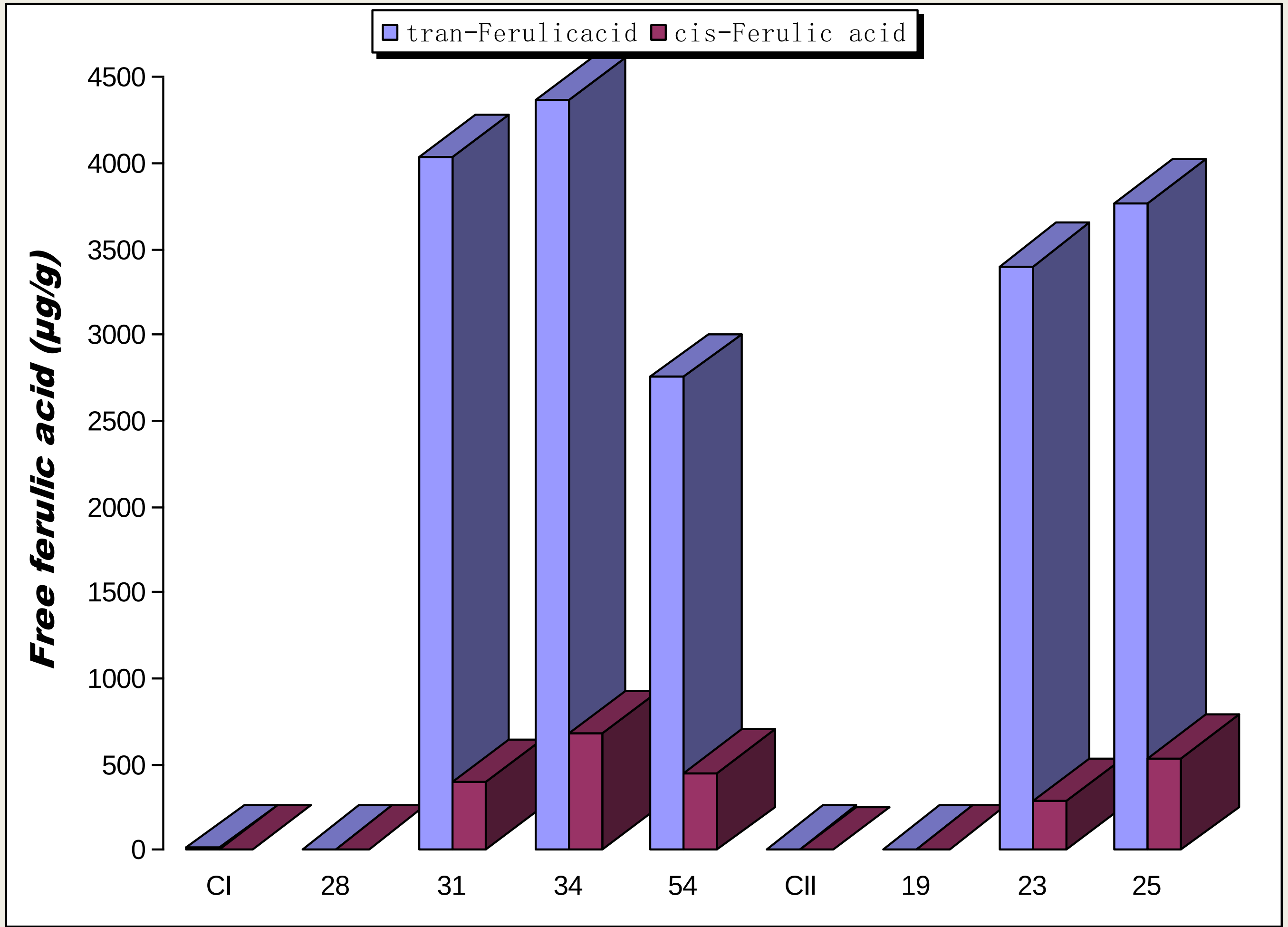

3c, .1N, Mix only
5.00kV x10k
5um


3d, .1N, Mix+HPH
5.00kV x10k
5um

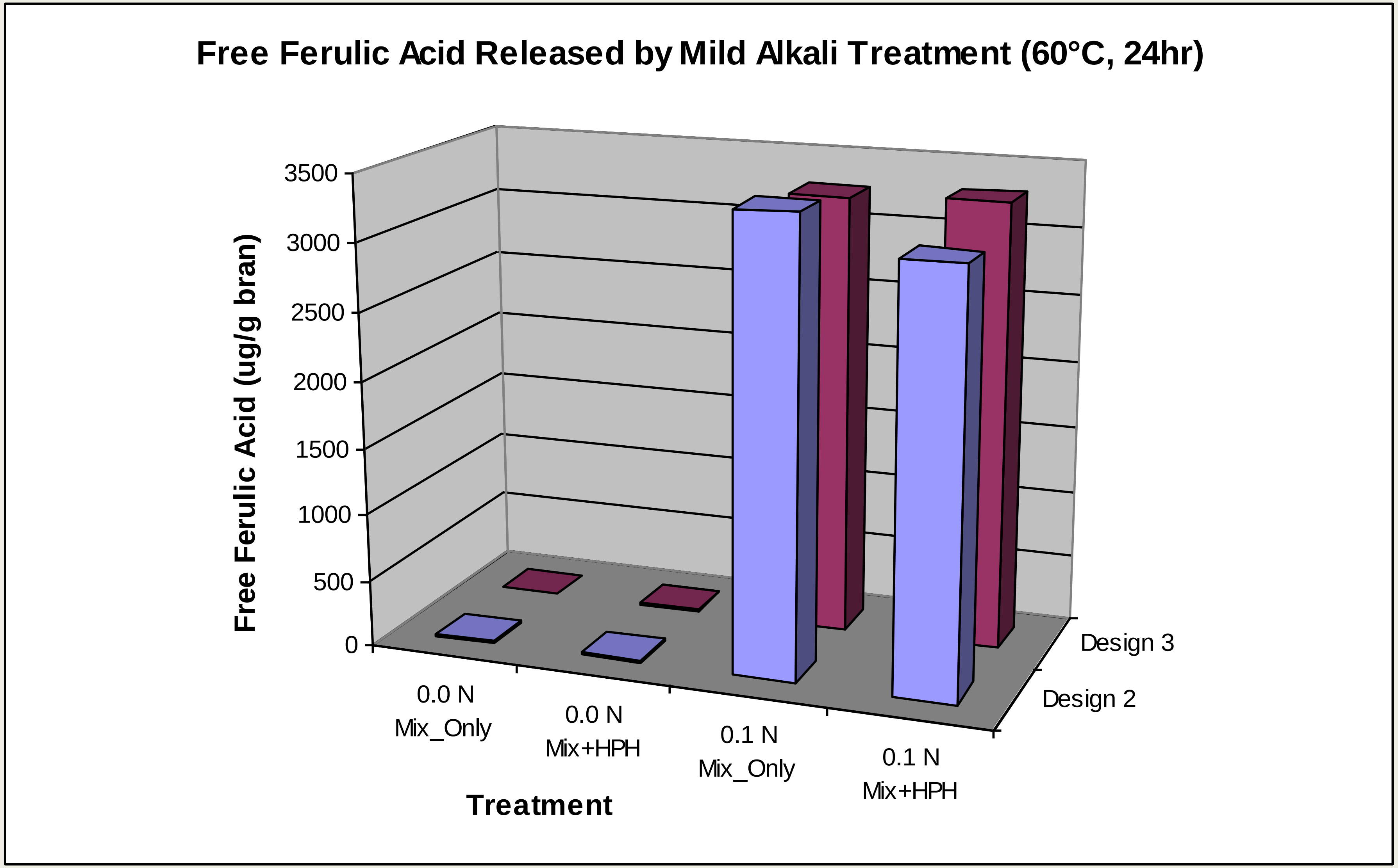

3c, .1N, Mix only
5.00kV x20k
2um


3d, .1N, Mix+HPH
5.00kV x20k
2um

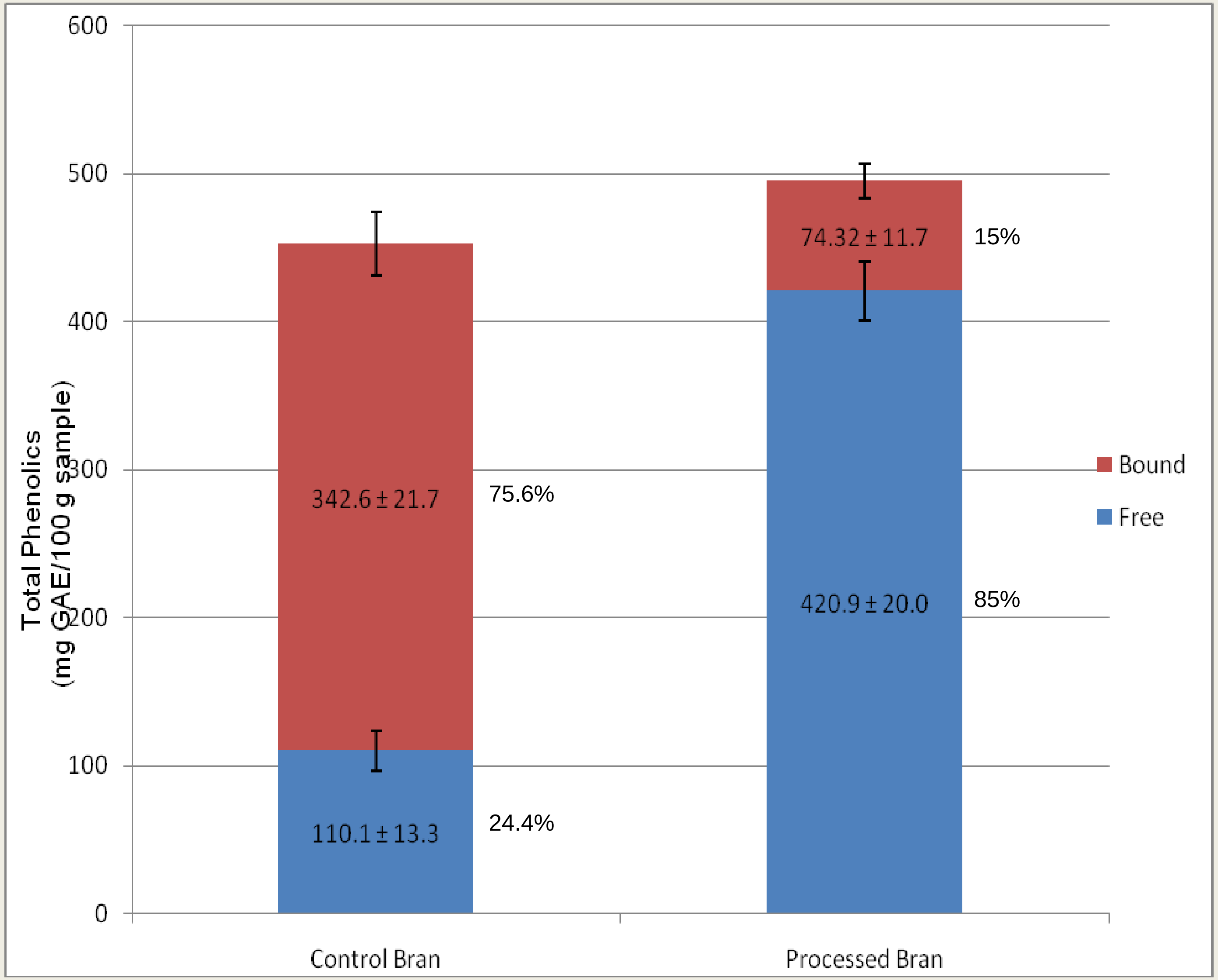
Effect of treatments on release of phenolics from wheat bran



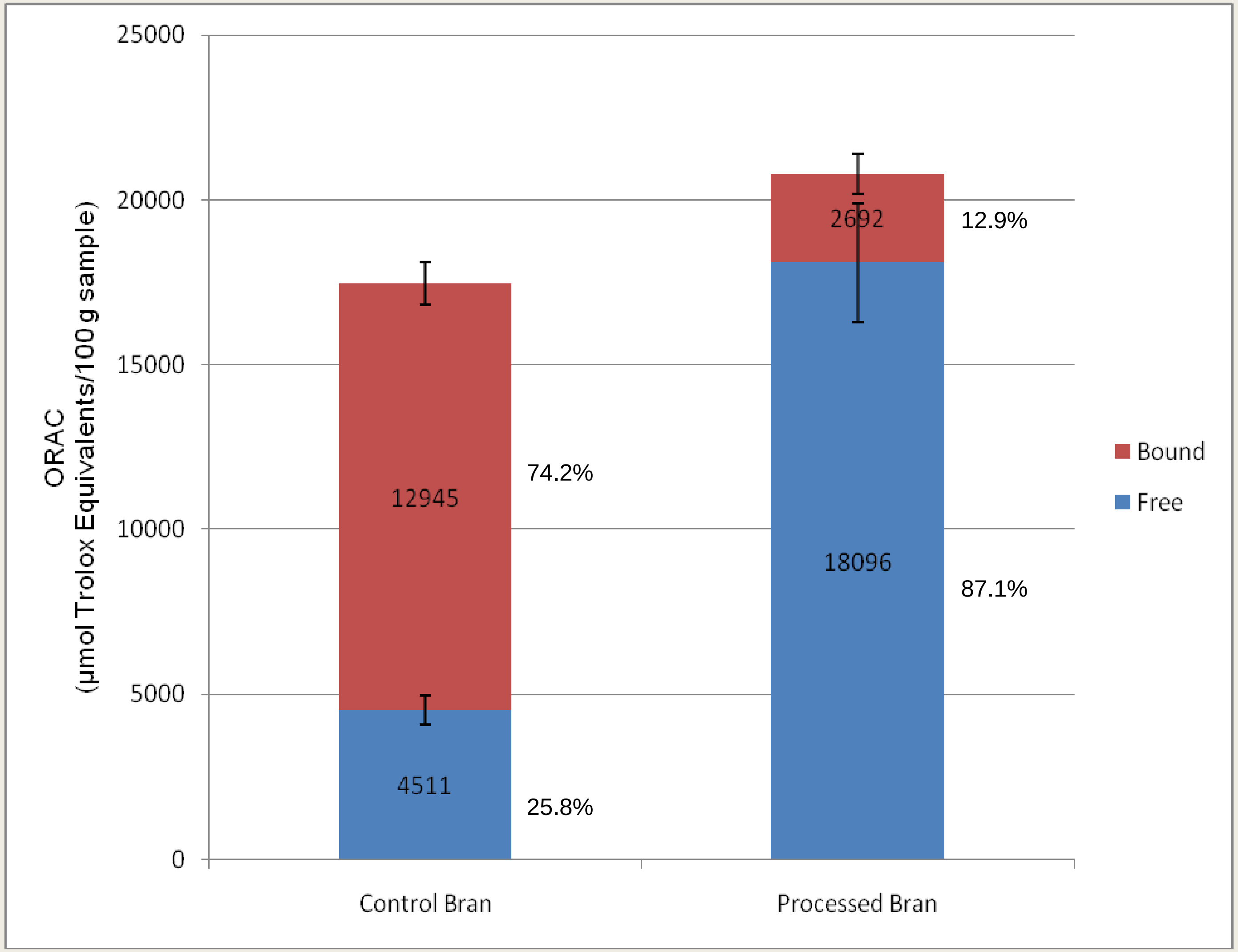
Bran Grind(mm)	0.2	0.2	0.2	0.2		0.5	0.5	0.5
NaOH Conc (*N*)	0.00	0.05	0.10	0.20		0.00	0.05	0.10
Soak time (hr)	6	6	6	24		6	15	6
Soak Temp (℃)	60	60	60	60		60	60	60
HPH (×1000psi)	22	22	22	22		22	22	22


Free Ferulic Acid Released by Mild Alkali Treatment (60°C, 24hr)
Free Ferulic Acid (ug/g bran)
3500
3000
2500
2000
1500
1000
500
0
0.0 N Mix_Only
0.0 N Mix+HPH
0.1 N Mix_Only
0.1 N Mix+HPH
Treatment
Design 3
Design 2

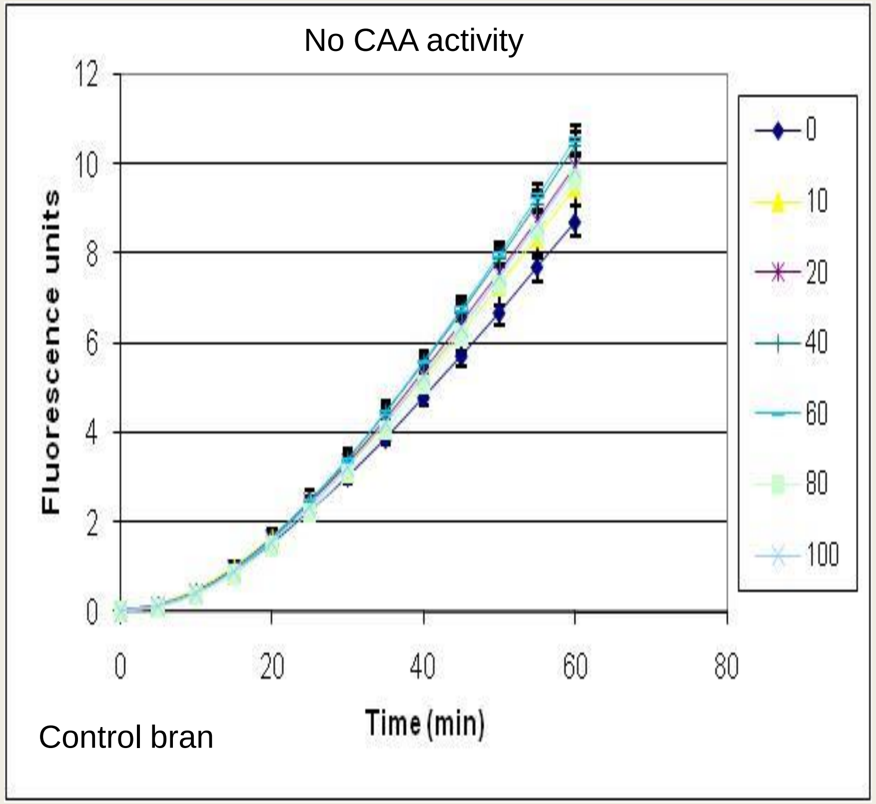
Total phenolic content of control bran and processed bran samples (mean ± SD, n = 3)

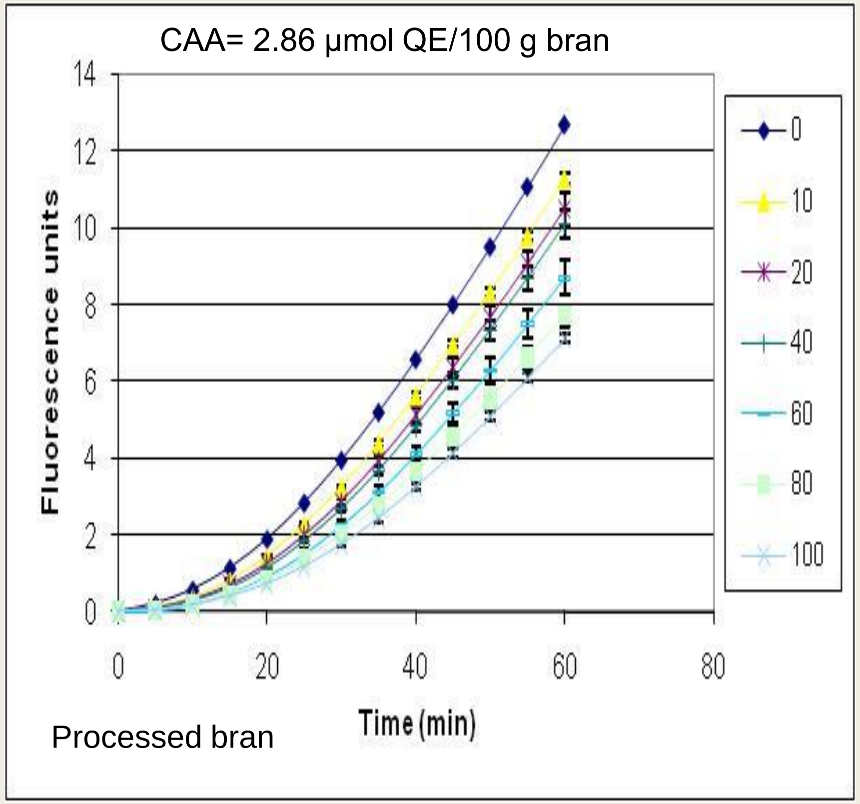


Total antioxidant activity of control bran and processed bran samples (mean ± SD, n = 3)

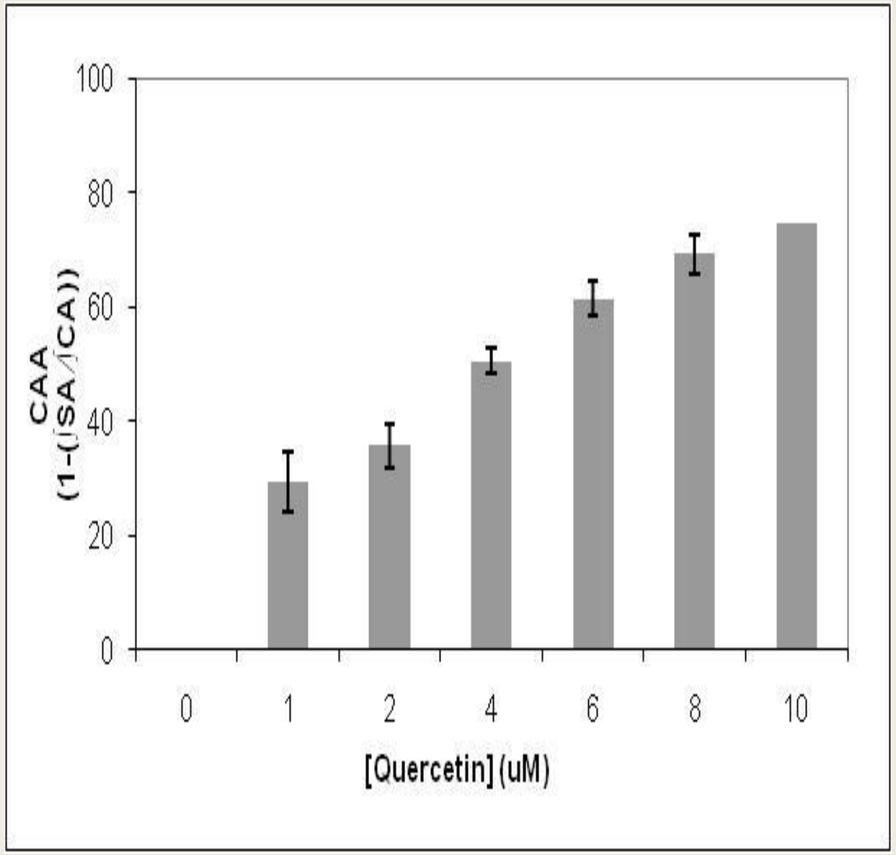


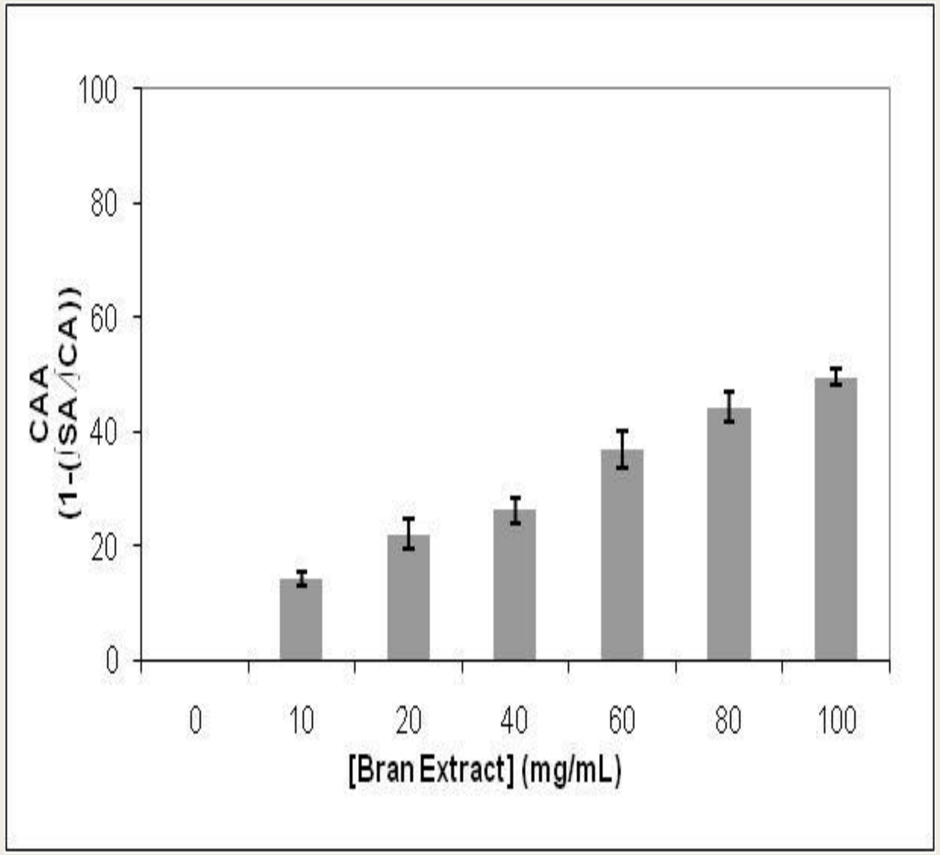
Cellular antioxidant activity (CAA) of control bran and processed bran samples (mean ± SD, n = 3)





Cellular antioxidant activity (CAA) of quercetin and processed bran samples when compared to the control (mean ± SD, n = 3)





THE ZUCKER DIABETIC FATTY (ZDF) RAT

- ✓ *Early-onset obesity due to a miss-sense mutation of the leptin receptor gene*

- ✓ *Displays*
  - ✓ Obesity
  - ✓ Hyperglycemia
  - ✓ Insulin resistance
  - ✓ Hyperlipidemia

- ✓ *Model for metabolic syndrome*

EXPERIMENTAL DIETS

- Basal diet was cornstarch-based (AIN-93G)

- The dietary groups were as follows:

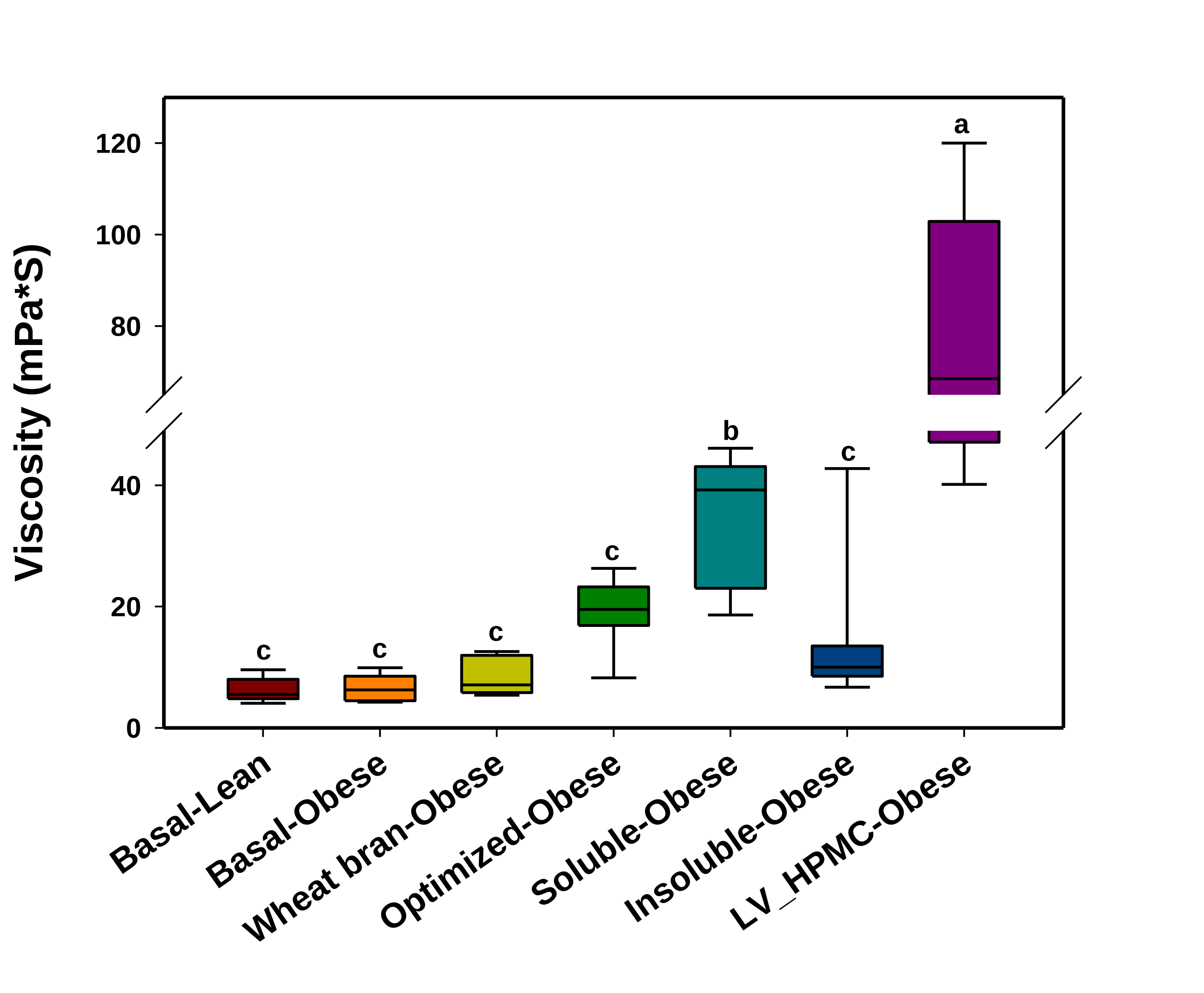
  - ✓ *Basal diet fed lean littermates (negative control) -* ***Basal_Lean***
  - ✓ *Basal diet fed ZDF rats (positive control) -* ***Basal_Obese***
  - ✓ *16.4 % Wheat bran-fed ZDF rats -* ***Wheat bran_Obese***
  - ✓ *22.9 % Optimized wheat bran-fed ZDF rats -* ***Optimized_Obese***
  - ✓ *27 % Soluble fraction of optimized wheat bran-fed ZDF rats -* ***Soluble_Obese***
  - ✓ *14.1% Insoluble fraction of optimized wheat bran-fed ZDF rats -* ***Insoluble_Obese***
  - ✓ *Low viscosity HPMC-fed ZDF rats (positive control for viscosity) -* ***LV HPMC_Obese***
    - HPMC (Hydroxypropyl methylcellulose)

- Diets were balanced for macronutrient composition

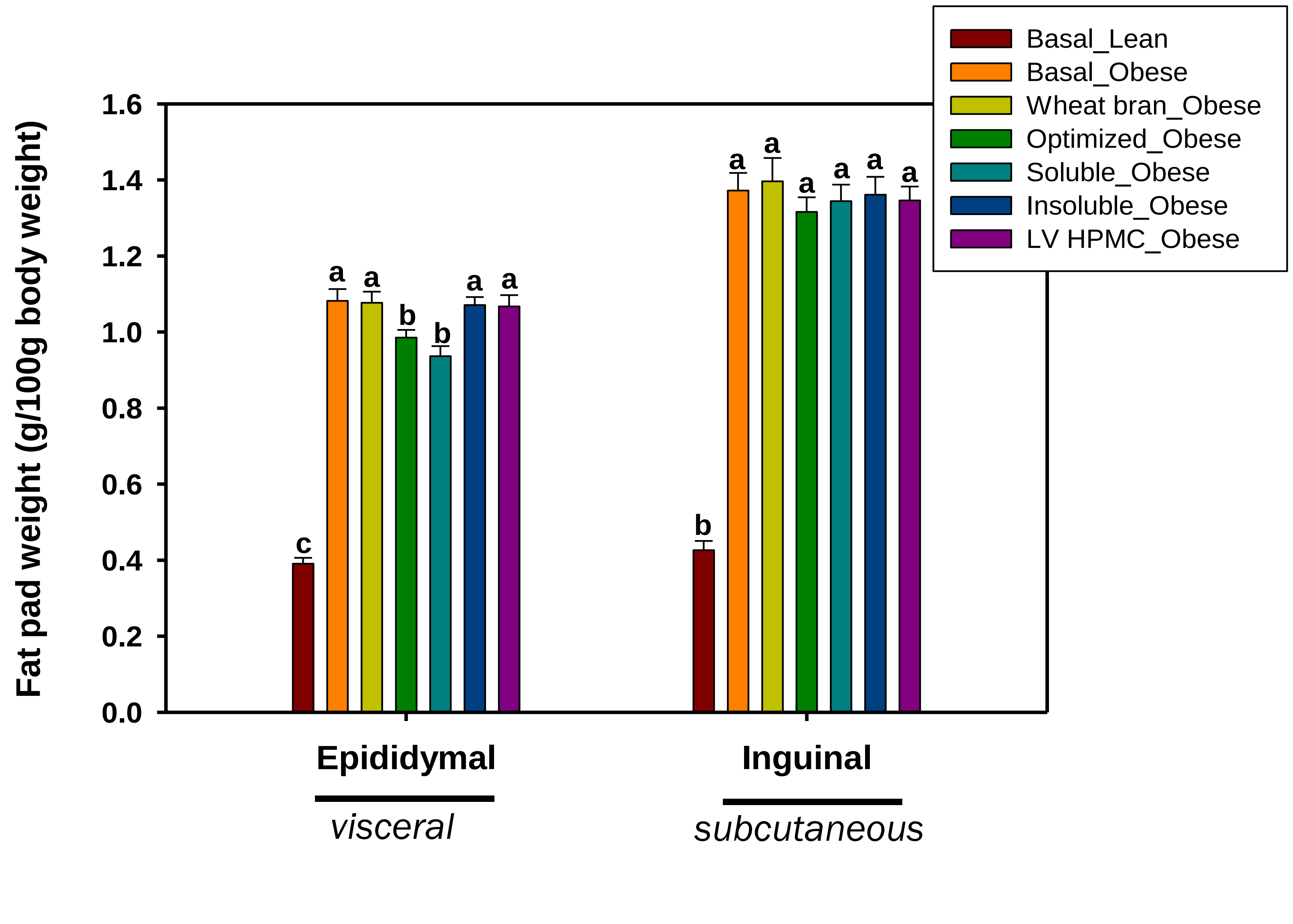
  55% Carbohydrate   20% protein
  8% Fiber   12% fat

- Diets fed for 3 weeks

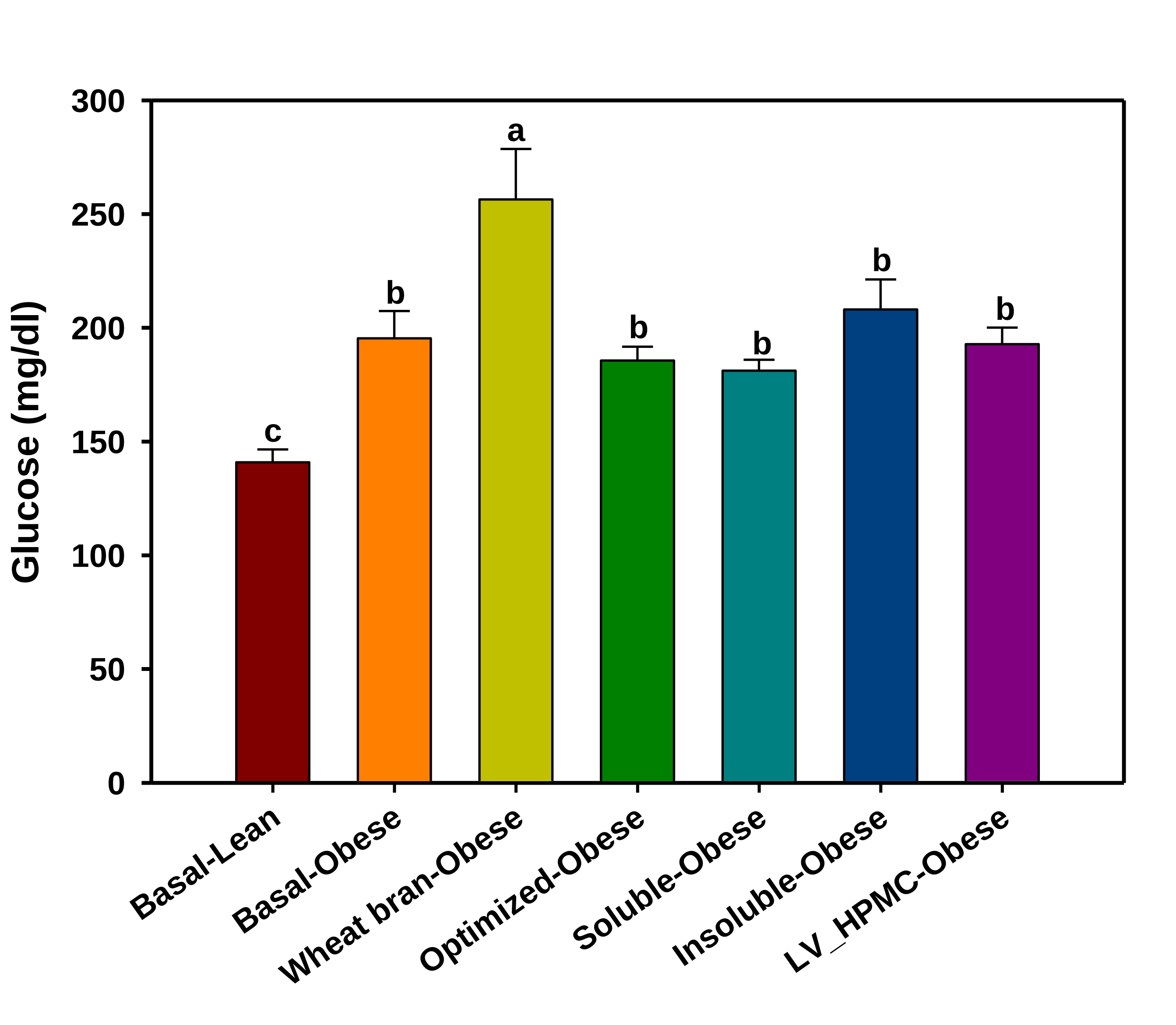
VISCOSITY OF SUPERNATANT OF INTESTINAL CONTENTS



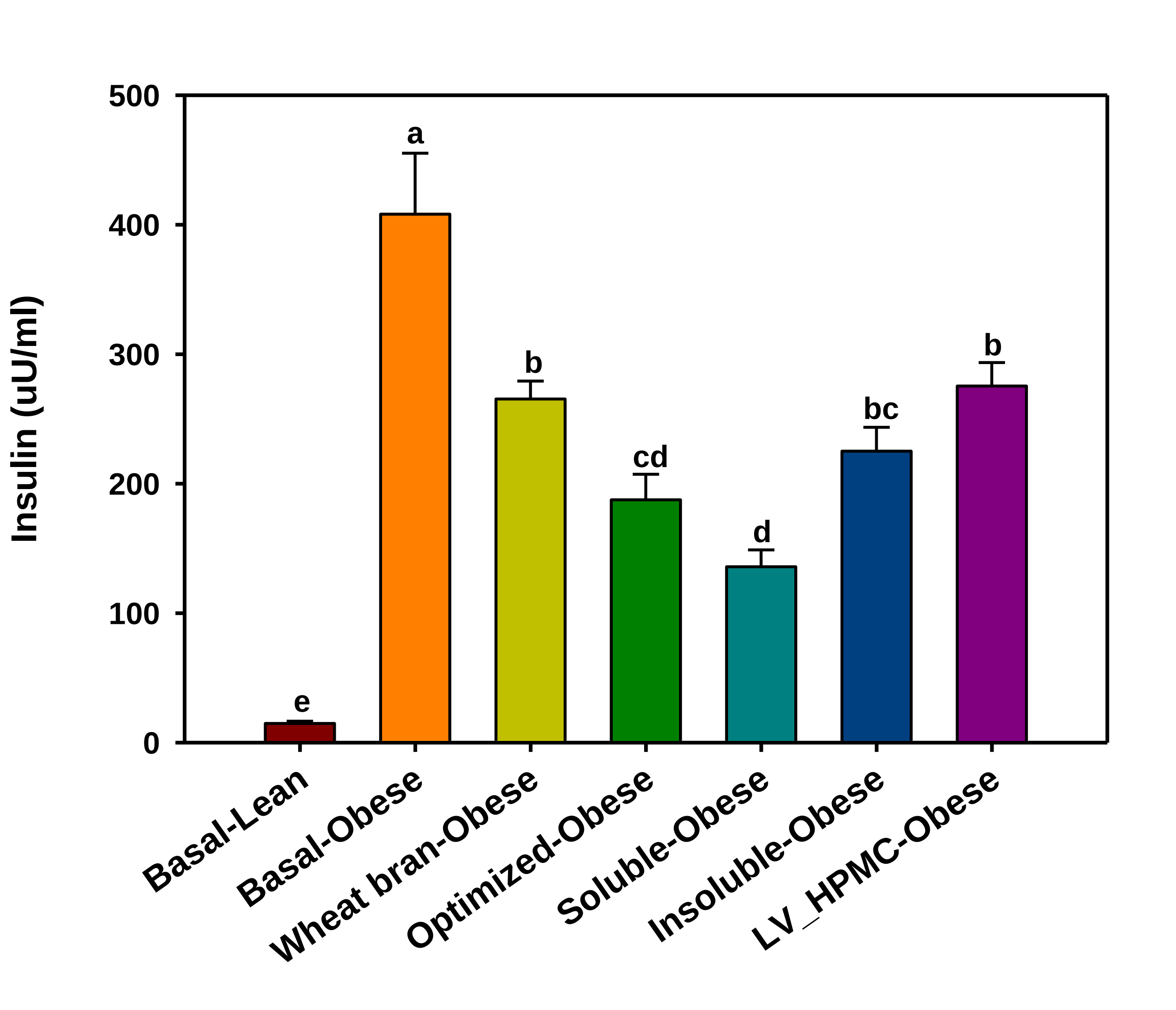
RELATIVE FAT PAD WEIGHT



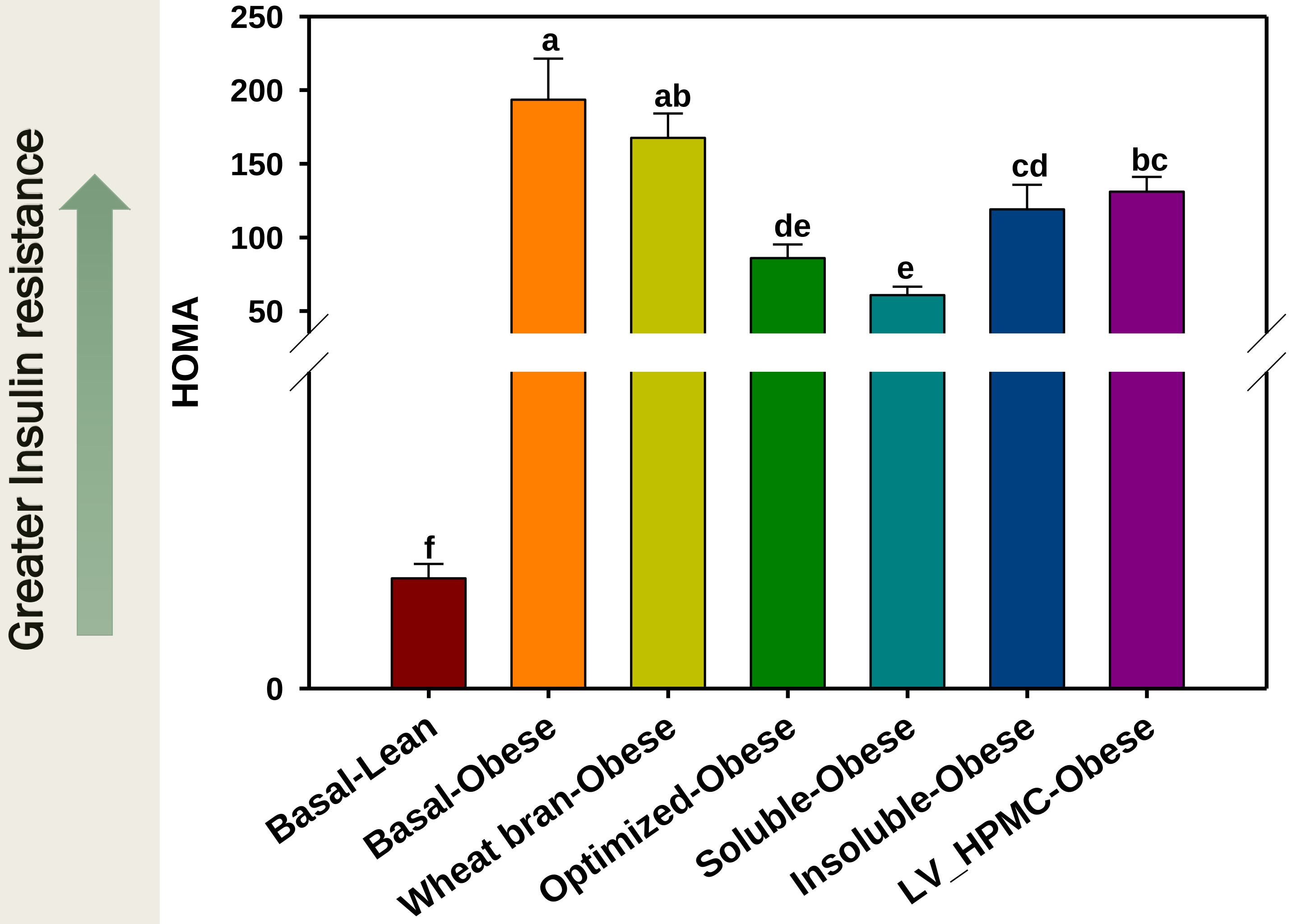
FASTING PLASMA GLUCOSE



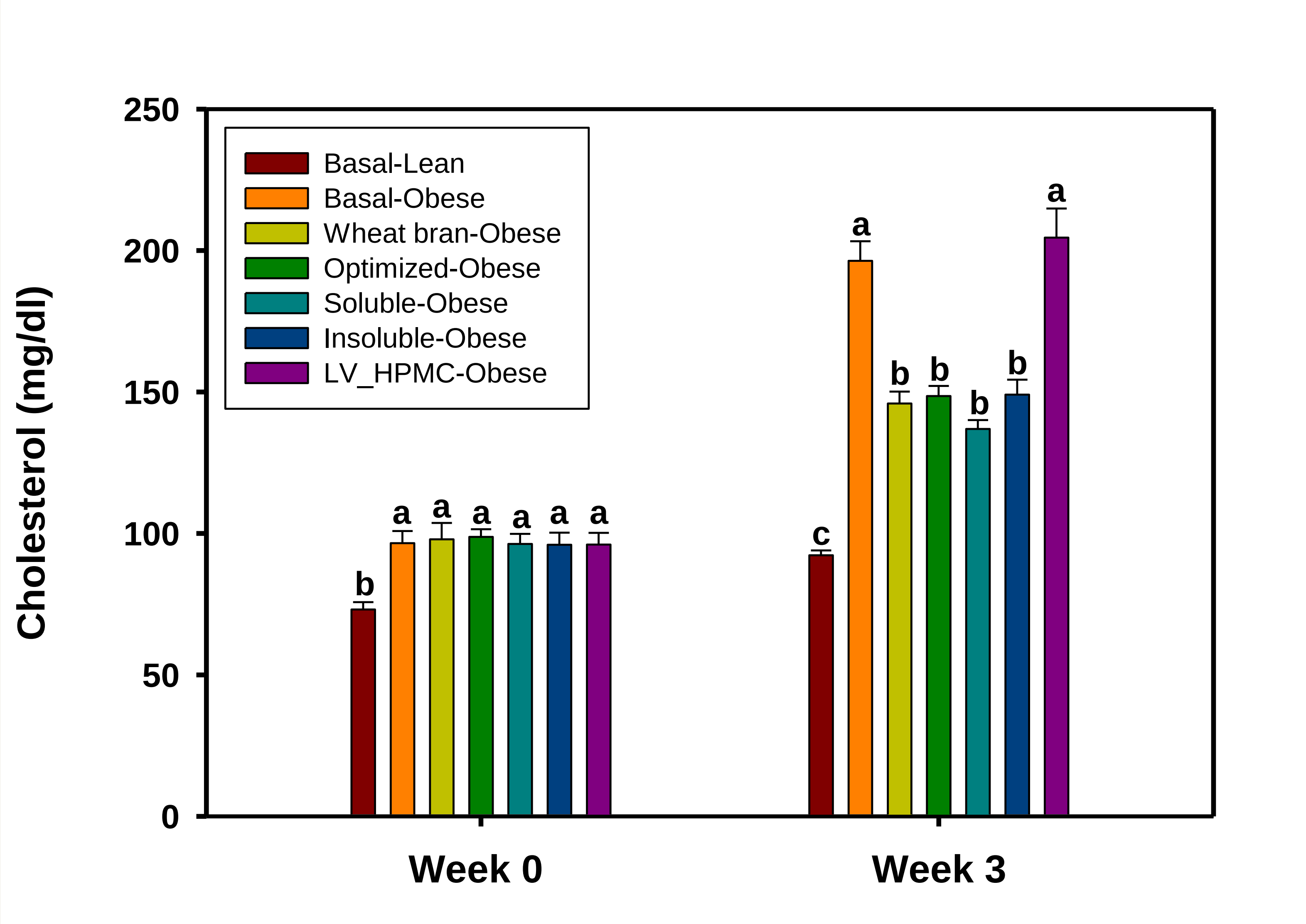
FASTING PLASMA INSULIN



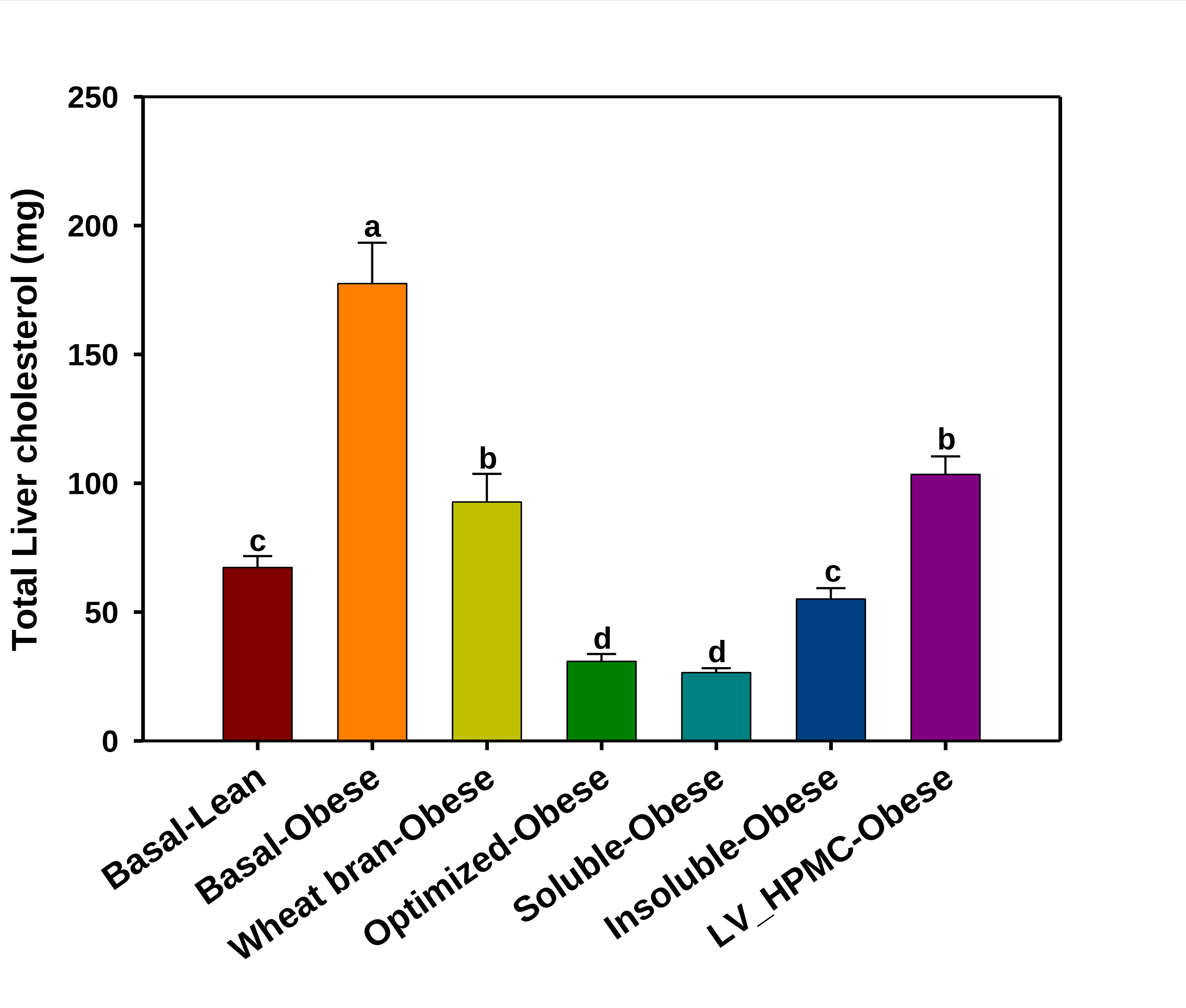
INSULIN RESISTANCE



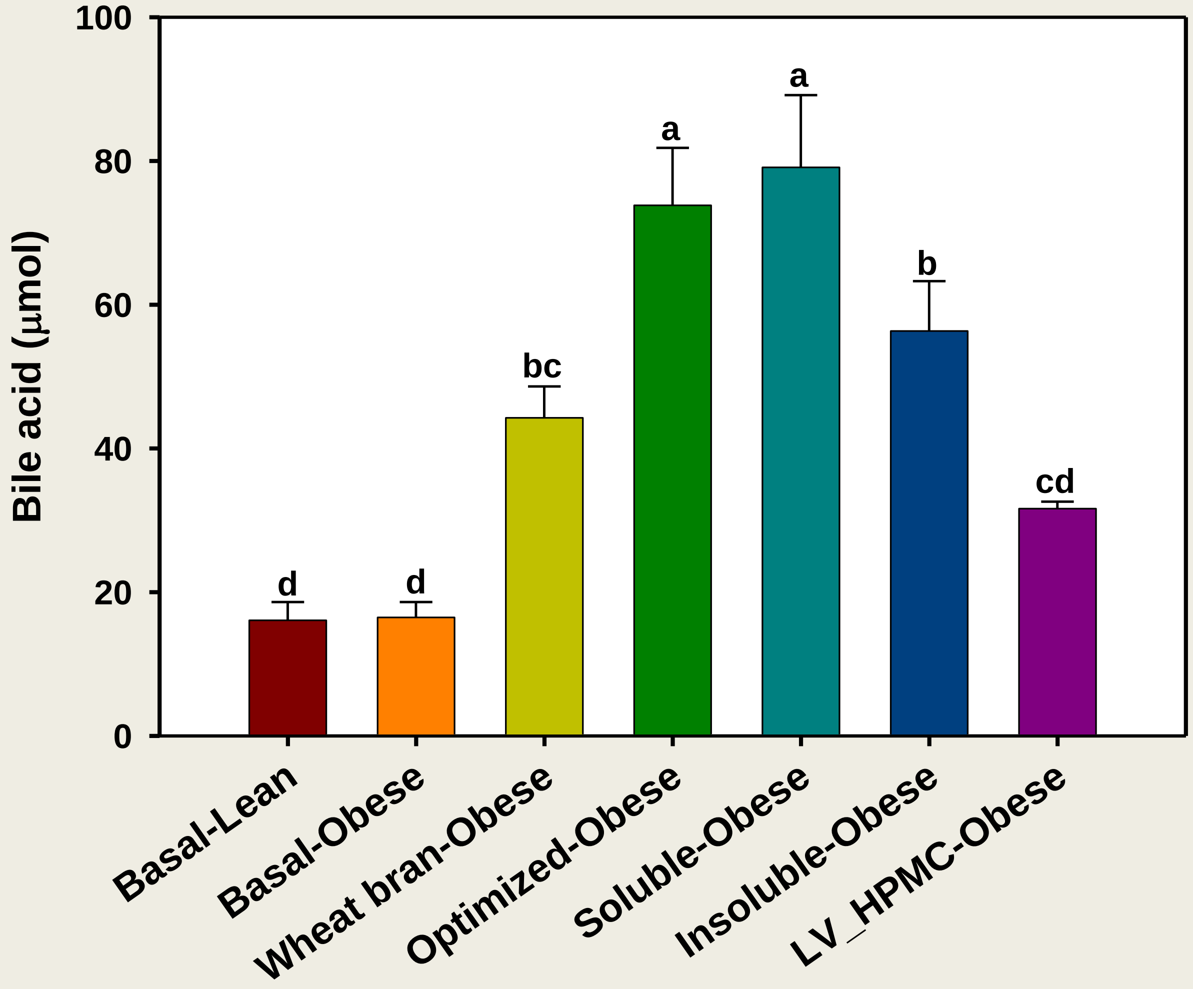
PLASMA CHOLESTEROL



LIVER CHOLESTEROL



FECAL BILE ACID EXCRETION



Objectives for Almond Hulls Utilization

- Analyze and understand the basic physical and chemical properties of different types of almond hulls
- Develop processes for effective and efficient extraction/separation of anti-oxidants and fibers
- Study the physiochemical properties of varieties of almond hulls after processing
- Extract antioxidative compounds and evaluate the antioxidant ability
- Optimize the processes based on feedback from analytical results
- Develop potential product concepts
- Plan Phase II work

Preliminary Almond Hall Characterization Process

Rinse

Three types of coarse almond hulls samples: Carmel, Nonpareil, Hardshell, were washed twice in cool tap small water (with small amount of shells and nuts, stalks removed).

Drying

The samples were then freezed in -20 °C for 24 hours and drying for 5 days until completely dried out.

Milling

The particle sizes of dried samples were broken down through a mechanical hammer mill.

Sieve

The fined particles were selected through 60 mesh (250um) lab sieves after being milled twice.

Analysis

Followed up with a series of composition analysis, physiochemical test and antioxidant ability analysis.

Almond Hulls Characterization

Types		Carmel	Nonpareil	Hardshell
Crude Protein	**%DM**	**6.63**	**5.71**	**3.45**
AD-ICP	%DM	1.46	1.38	1.21
ND-ICP w/ SS	%DM	1.67	1.39	1.24
Protein Sol.	%CP	40.47	43.41	38.23
Sugar (WSC)	**%DM**	**20.97**	**31.73**	**36.45**
ADF	%DM	28.66	18.06	21.79
aNDF	**%DM**	**34.72**	**22.35**	**24.85**
Lignin (Sulfuric Acid)	%DM	10.45	6.72	8.20
Lignin	%NDF	30.57	31.30	33.37
Starch	%DM	0.06	0.01	0.01
Fat (EE)	**%DM**	**4.08**	**2.66**	**2.14**
Ash	**%DM**	**6.13**	**7.92**	**5.92**

Preliminary Polyphenol Compounds Extraction Process

5.00g Almond hull sample

↓

Magnetic stirring at 1,150 rpm for 30 minutes

↓

Extraction with ultrasound for 30 min at room temperature

↓

Centrifuge at 2,000 x g at 10 ℃ for 10 min

↓

Dispose supernatants

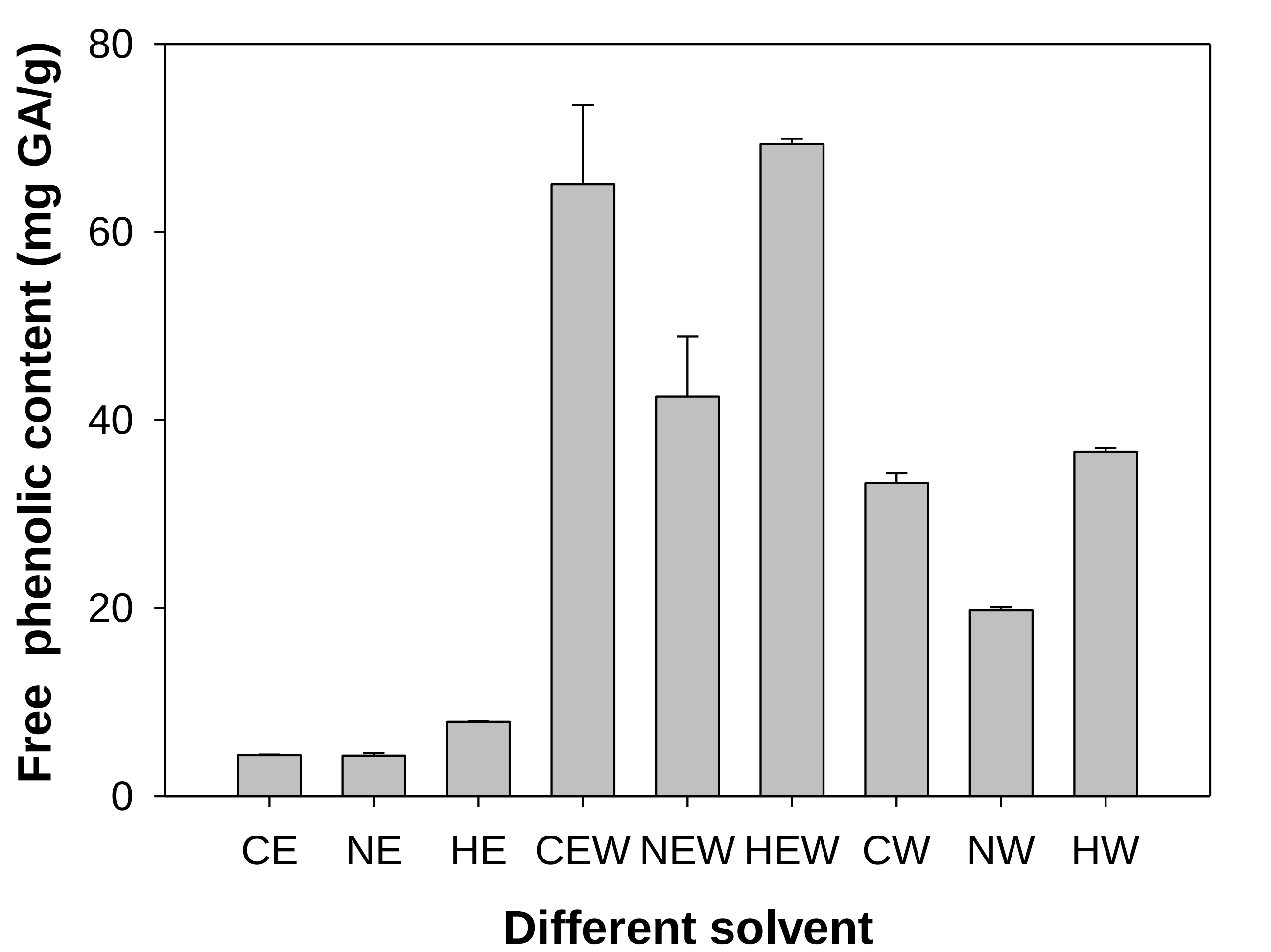
↓

Soluble fraction (Free phenolic compounds)

Extraction agent 1: 100 mL of ethyl alcohol (E) added
Extraction agent 2: 50 mL ethyl alcohol 50 mL distilled water (EW) (1:1).
Extraction agent 3: 100 mL of distilled water (W)

Phenol Content Determination

- The phenolic content of almond hull extract was determined using the spectrophotometric method. A 0.5 mL sample (1 mg/ml) was mixed with 0.5 mL of Folin-Ciocalteu's phenol reagent (1:2 dilution). After 5 min, 5 mL of a 7.5% Na_2CO_3 solution was added to the mixture. The solution was left to stand at room temperature ($23 \pm 2\,^\circ C$) for 30 min, and the absorbance was measured at 760 nm using a spectrophotometer against a blank sample.
- The phenolic content was calculated using a calibration curve for garlic acid. The results are expressed as the gallic acid equivalent per gram of dry weight of extract (mg of GAE/g of extract). All samples were analyzed in triplicate.

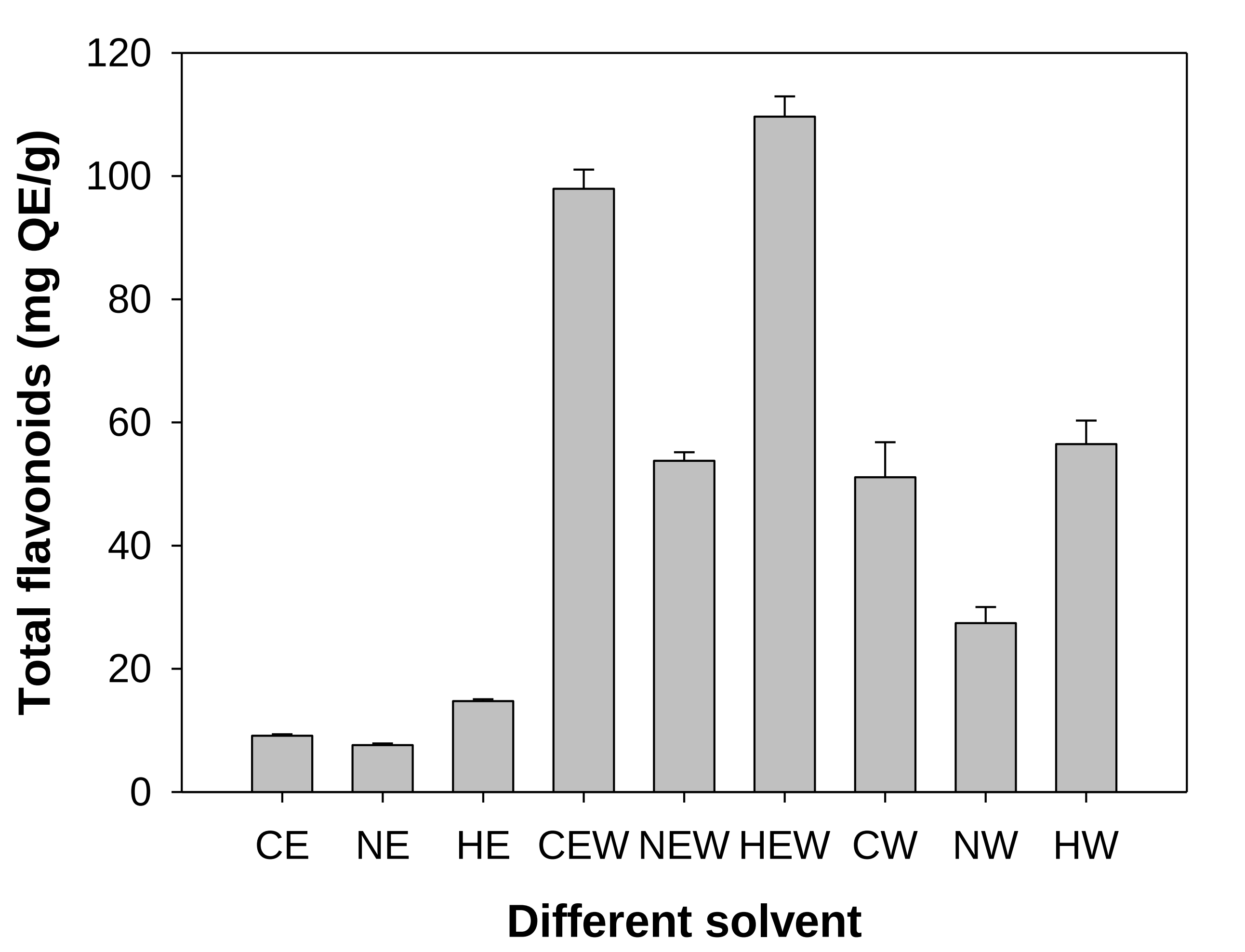


INFLUENCE OF DIFFERENT SOLVENT ON FREE PHENOLIC CONTENT OF ALMOND HULL EXTRACT

CE – Carmel extracted with ethanol
NE – Nonpareil with ethanol
HE – Hardshell with ethanol
CEW – Carmel with ethanol and water
NEW – Nonpareil with ethanol and water
HEW – Harshell with ethanol and water
CW - Carmel with water
NW – Nonpareil with water
HW – Harshell with water

Total flavonoids content (TFC)

- TFC analysis was colorimetrically performed using following the method. Briefly, 0.3 mL of extracts, 3.4 mL of 30% methanol, 0.15 mL of $NaNO_2$ (0.5 M) and 0.15 mL of $AlCl_3.6H_2O$ (0.3 M) were mixed. After 5 min, 1 mL of NaOH (1 M) was added. The solution was mixed well and the absorbance was measured against the reagent blank at 506 nm. The standard curve for total flavonoids was made using quercetin standard solution (0 to 100 mg/L) under the same procedure as earlier described. The total flavonoids were expressed as milligrams of quercetin equivalents per g of dried fraction.



INFLUENCE OF DIFFERENT SOLVENT ON TOTAL FLAVONOIDS CONTENT OF ALMOND HULL EXTRACT

CE – Carmel extracted with ethanol
NE – Nonpareil with ethanol
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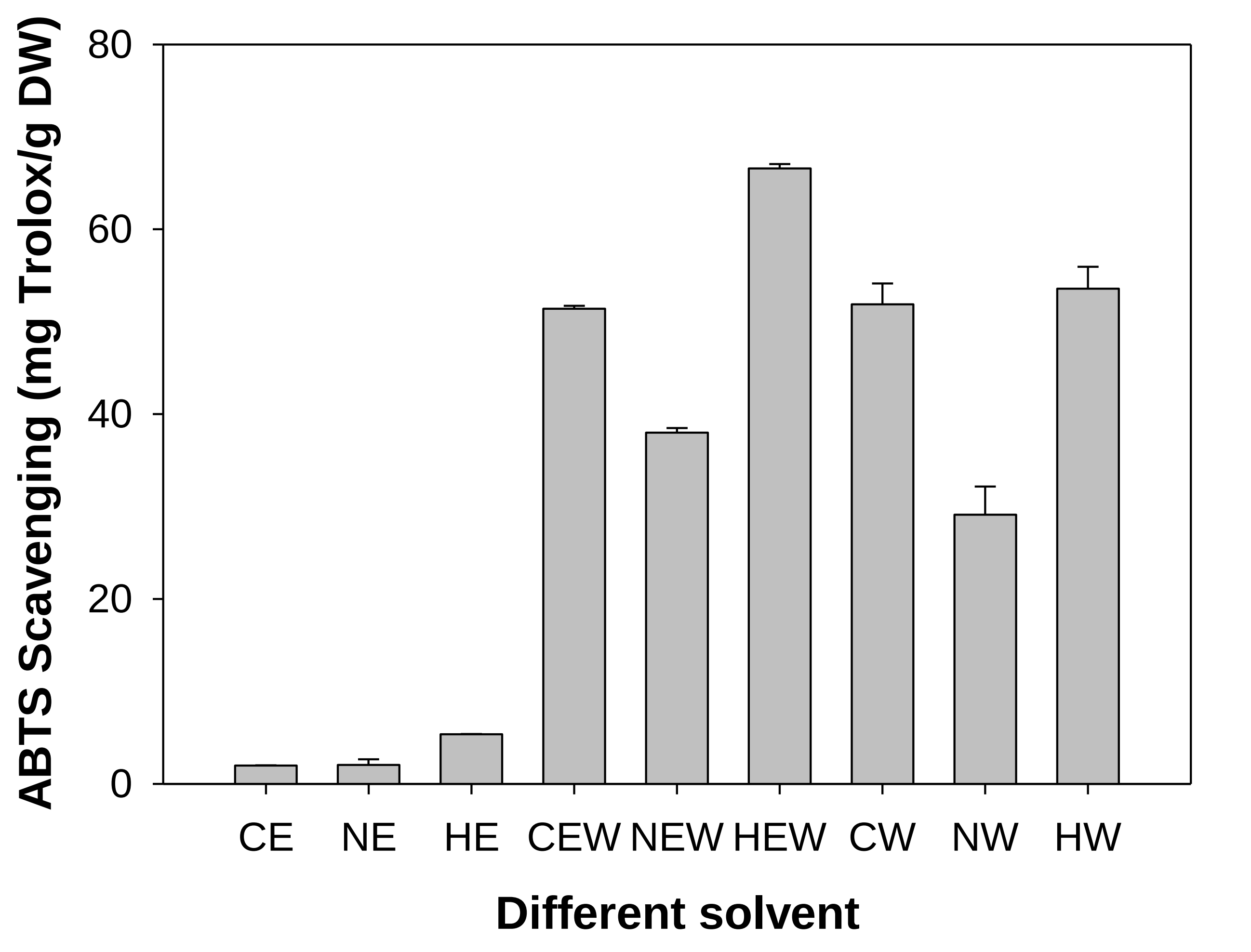
CEW – Carmel with ethanol and water
NEW – Nonpareil with ethanol and water
HEW – Harshell with ethanol and water
CW - Carmel with water
NW – Nonpareil with water
HW – Harshell with water

$ABTS^+$ radical scavenging assay to determine the antioxidant capacity

- The stock solution included 7.4 mM ABTS solution and 2.6 mM potassium persulphate solution. The working solution was prepared by mixing the two stock solutions in equal quantities and allowing them to react for 12 h at room temperature in the dark. The solution was diluted by mixing 1 mL of ABTS solution with 60 mL methanol to obtain an absorbance of 1.1 ± 0.02 units at 734 nm. Fresh ABTS solution was prepared for each assay. Extracts (150 uL) were mixed with 2850 L of ABTS and allowed to react at 20 ºC until a steady absorbance was reached. Methanol was used as the control. The Genesys-5 UV/Vis spectrophotometer was blanked with methanol, and the decrease in absorbance due to antioxidants was recorded at 734 nm. The antioxidant activity was calculated as mol of Trolox equivalents (TE) per g sample DW from a standard curve developed with Trolox.

INFLUENCE OF DIFFERENT SOLVENT EXTRACTED ALMOND HULL ANTIOXIDANTS ON ABTS SCANVENGING ABILITY



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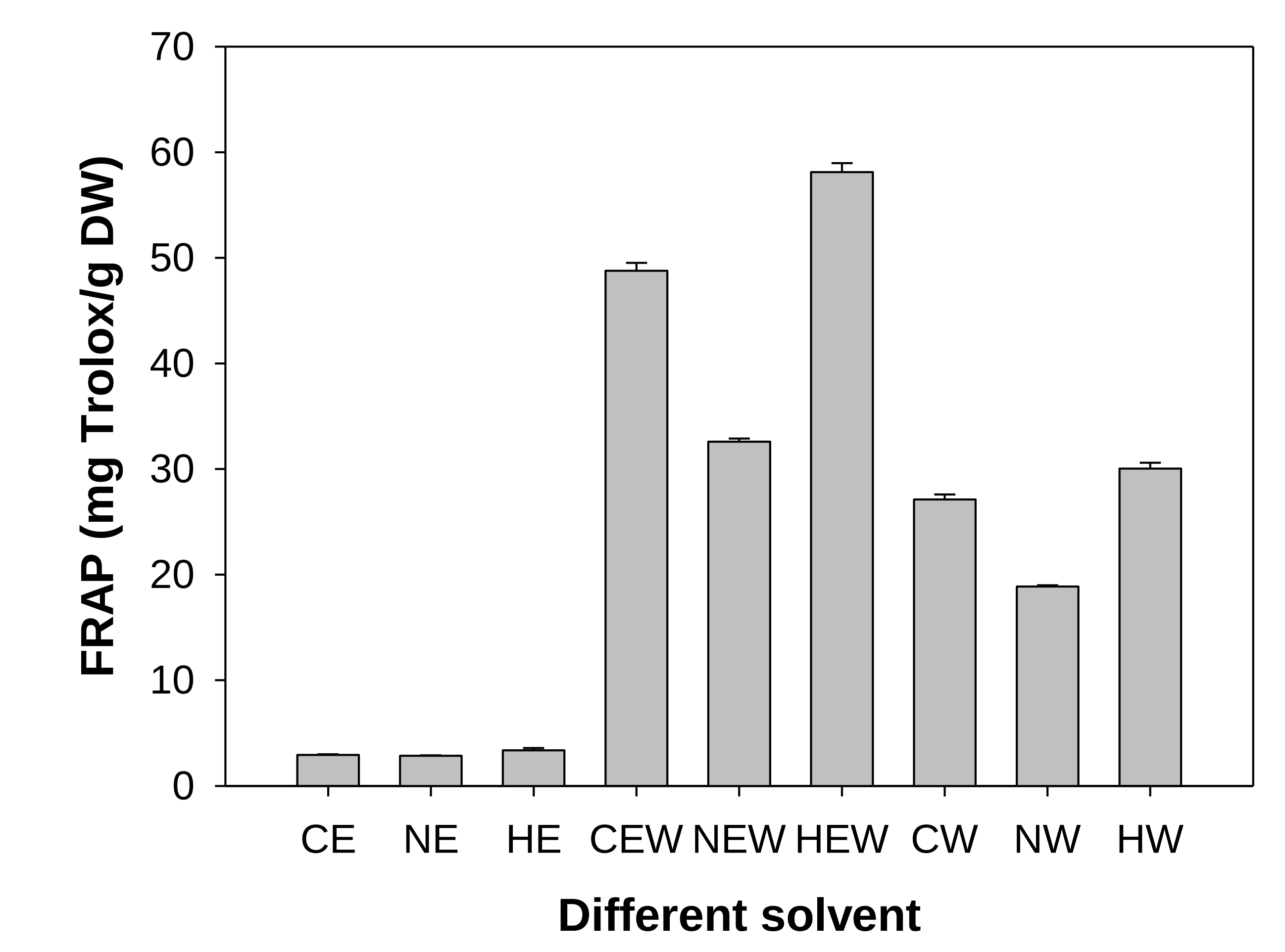
CEW – Carmel with ethanol and water
NEW – Nonpareil with ethanol and water
HEW – Harshell with ethanol and water
CW - Carmel with water
NW – Nonpareil with water
HW – Harshell with water

FRAP assay to determine the antioxidant capacity

- The FRAP reagent contained 2.5 ml of a 10 mmol/L TPTZ (2,4,6- tripyridy-s-triazine, Sigma) solution in 40 mmol/L HCl plus 2.5 ml of 20 mmol/L $FeCl_3$ and 25 ml of 0.3 mol/L acetate buffer, pH 3.6 and was prepared freshly and warmed at 37° C. Aliquots of 0.3 mL sample and 2.7 ml FRAP reagent and the absorbance of reaction mixture at 593 nm was measured spectrophotometrically after incubation at 37° C for 10 min. The Trolox was used as the standard solution. The final result was expressed as the concentration of antioxidants having a ferric reducing ability equivalent to that of 0.1 mg Trolox/g dry weight. Adequate dilation was needed if the FRAP value measured was over the linear range of standard curve. ($y=12.4138x+0.1435$, $R^2= 0.9996$).

EFFECT OF DIFFERENT POLARITY SOLARITY SOLVENT EXTRACTED ALMOND HULL ANTIOXIDANTS ON FERRIC REDUCING/ANTIOXIDANT POWER



CE – Carmel extracted with ethanol
NE – Nonpareil with ethanol
HE – Hardshell with ethanol
CEW – Carmel with ethanol and water
NEW – Nonpareil with ethanol and water
HEW – Harshell with ethanol and water
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NW – Nonpareil with water
HW – Harshell with water

Comparison of phenolic contents among different agricultural by-products

By-product	Total phenolic levels (mg/g dw)	Total flavonoids level (mg/g dw)	References
Almond	*See next slide*		
Apple peels	33.42	22.99	Wolfe and Liu (2003)
Avocado peels	44.06		Gomez-Caravaca et al. (2015)
Buckwheat hulls	3.33	1.38	Quettier-Deleu et al. (2000)
Cornelian cherry pulp	26.80		Agourram etal. (2012)
Cornelian cherry seeds	33.30		Agourram etal. (2012)
Dog rose pulp	16.70		Agourram etal. (2012)
Dog rose seeds	16.30		Agourram etal. (2012)
Dried apple pomace	3.31	0.99	Rana et al. (2015)
Dried coconut husk	35.5-45.5	41.6-42.4	Valadez-Carmona et al. (2016)
Grape marcs	6.6-16.3		Agourram etal. (2012)
Grape seeds	37.40		Babbar et al. (2011)
Leek leaves	6.40		Agourram etal. (2012)
Pomegranate pomace	8.70		Agourram etal. (2012)
Potato peels	4.2-8.9		Agourram etal. (2012)
Potato pulps	0.8-4.2		Agourram etal. (2012)

Polyphenols and flavones in almond

Results from the literature

Source	Total polyphenol (mg/g DW)	Total flavone (mg/g DW)	References
Almond skin	0.58-1.77	0.16-0.53	Bradley et al, 2010; Pasqualone et al, 2018
Almond hull (Ethanol)	25.2-72.1	ND	Pinelo et al, 2004
Almond hull (Methanol)	10.6-41.2	ND	Pinelo et al, 2004

Our results (almond hulls)

Almond species	Free polyphenol mgGA/g	Total flavones mg QE/g
Carmel	65.11±2.38	97.92±3.14
Hardshell	69.35±0.56	109.66±3.26
Nonpareil	42.49±1.41	53.78±1.38

Preliminary processes to produce functional fibrous ingredients

Dispersion

Mix 100 g powders produced from the processing described previously with 1,000 mL distilled water. Then disperse the liquid at 10,000 rpm for 3 min with an immersion homogenizer.

Homogenization

Homogenize the sample with microfluidizer processer under 11,300 psi for 1 and 2 passes.

Drying

Freeze the sample in -20 ^{o}C for 14 hours and drying for 4-5 days until completely dried out.

Milling

The crunchy and flaky samples were milled and fine particles were selected through 60 mesh lab sieves.

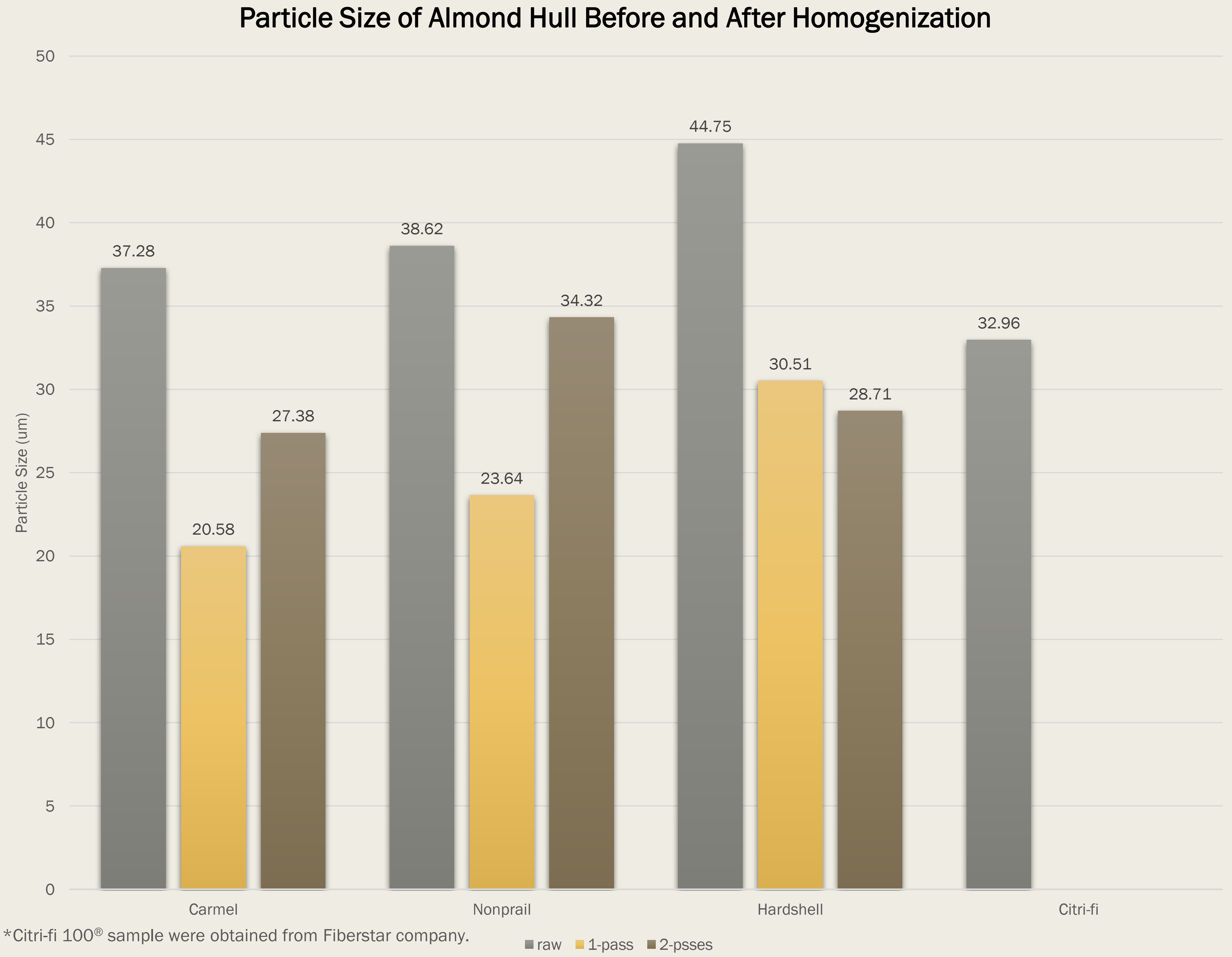
Analysis

Several physiochemical properties tests were performed to explore potential usage of this by-product.

Physiochemical Properties

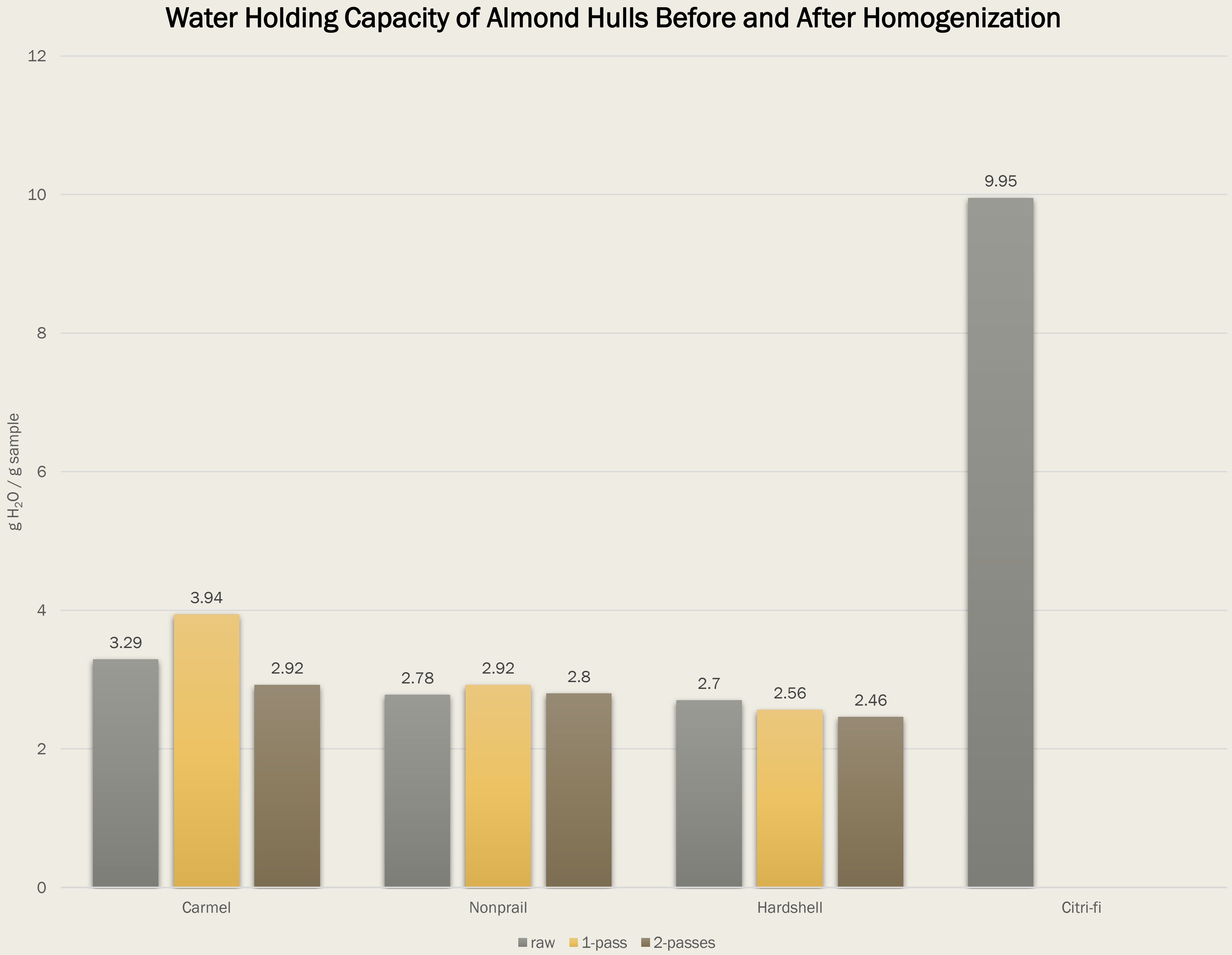
- Particle size
- Water-holding capacity (WHC)
- Oil-binding capacity (OBC)
- Emulsifying capacity
- Emulsifying stability

Laser Particle Size Analysis



*Citri-fi 100® sample were obtained from Fiberstar company.

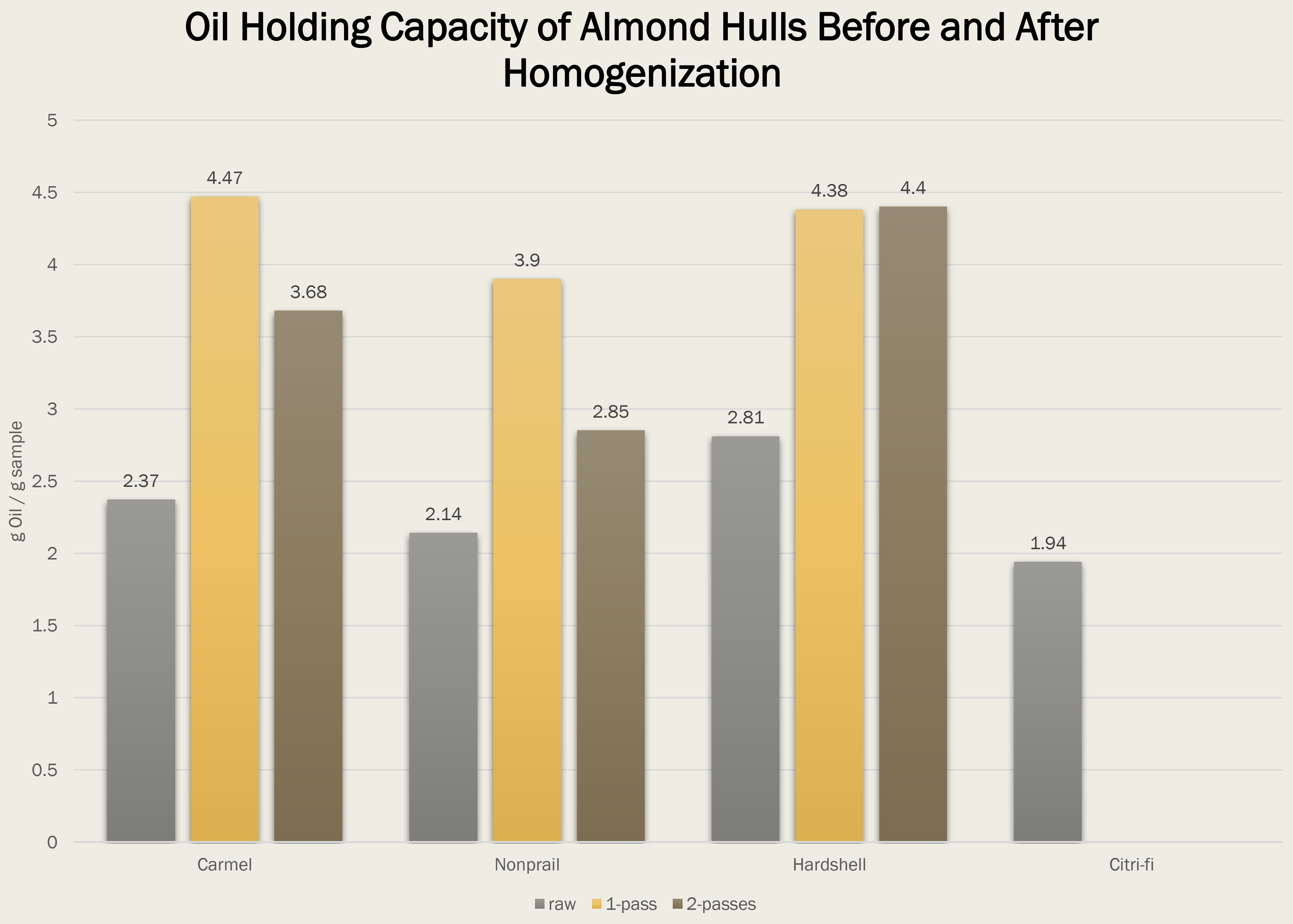
Water Holding Capacity (AACC 56-30)



Oil Holding Capacity

0.5 g sample + 5mL oil, vortex for 2 min, centrifuge in 2,000 g for 30 min, discard the supernatants.

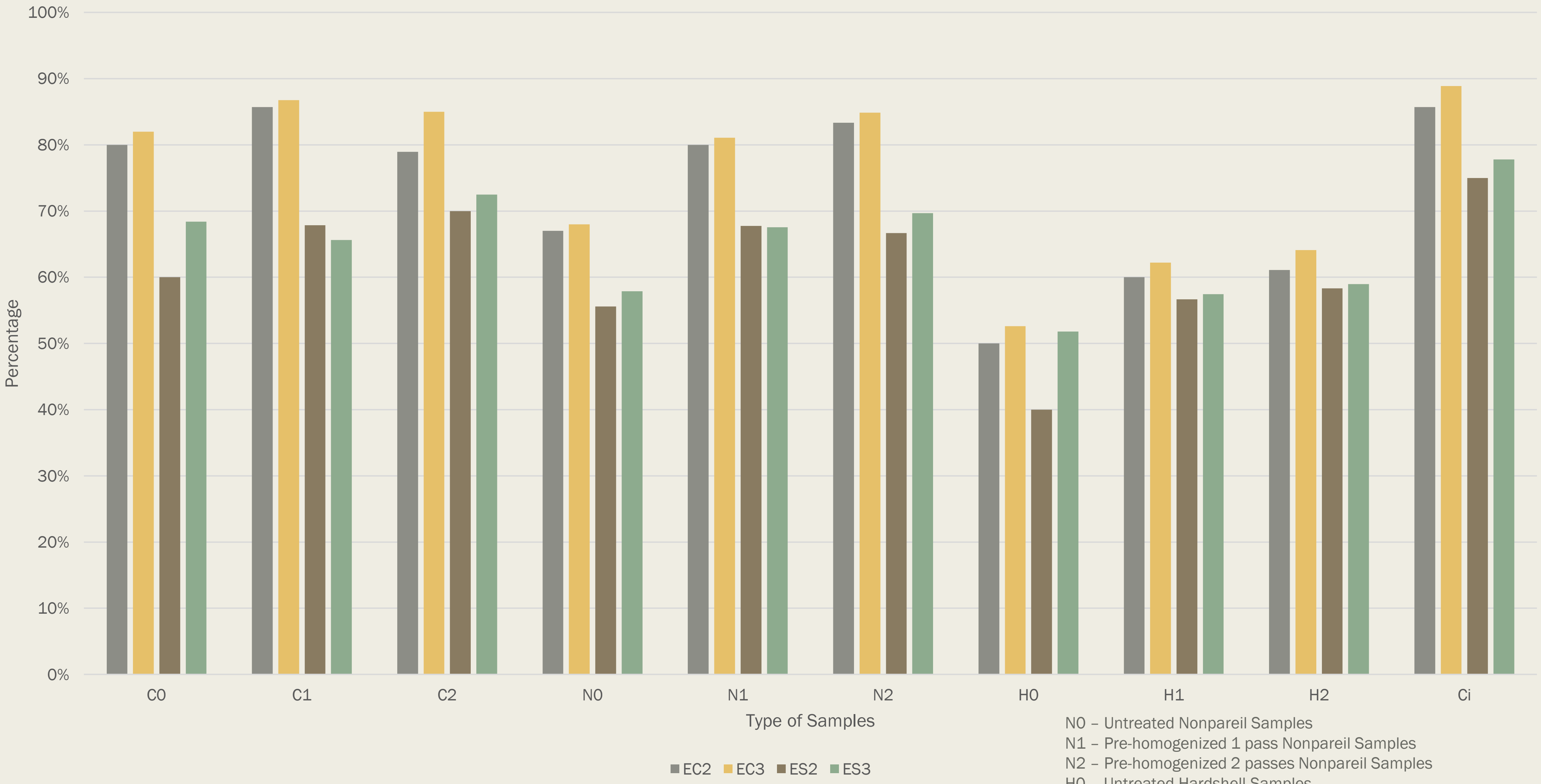
OHC (%) = (final weight – 0.5g – centrifuge tube weight)/0.5g



Emulsifying Capacity (EC) & Emulsifying Stability (ES) Tested For the Almond Hulls Powders Before Homogenization

- (1.5% concentration) Add 1.8 g raw almond hulls powders produced by step 1 into 60 mL distilled water, vortex and add 60 mL oil. Dispersed with 10,000 rpm for 3min, homogenized under 11,3000 psi for 2 and 3 passes. Centrifuged with 500 g for 10 min, measure the height of each layer in the emulsifying system.
- EC = $\frac{height\ of\ emulsifying\ layer}{total\ height}$
- Heat in 80 ^{o}C water bath for 30 min, cool down to 25 ^{o}C, centrifuge with 500 g for 10 min again, then measure the height of layers. Emulsifying stability is calculated as emulsifying capacity.
- ES = $\frac{height\ of\ emulsifying\ layer}{total\ height}$

Emulsifying Capacity and Emulsifying Stability of Almond Hulls Samples, Pre-homogenized Almond Hulls Samples and Citri-fi Product

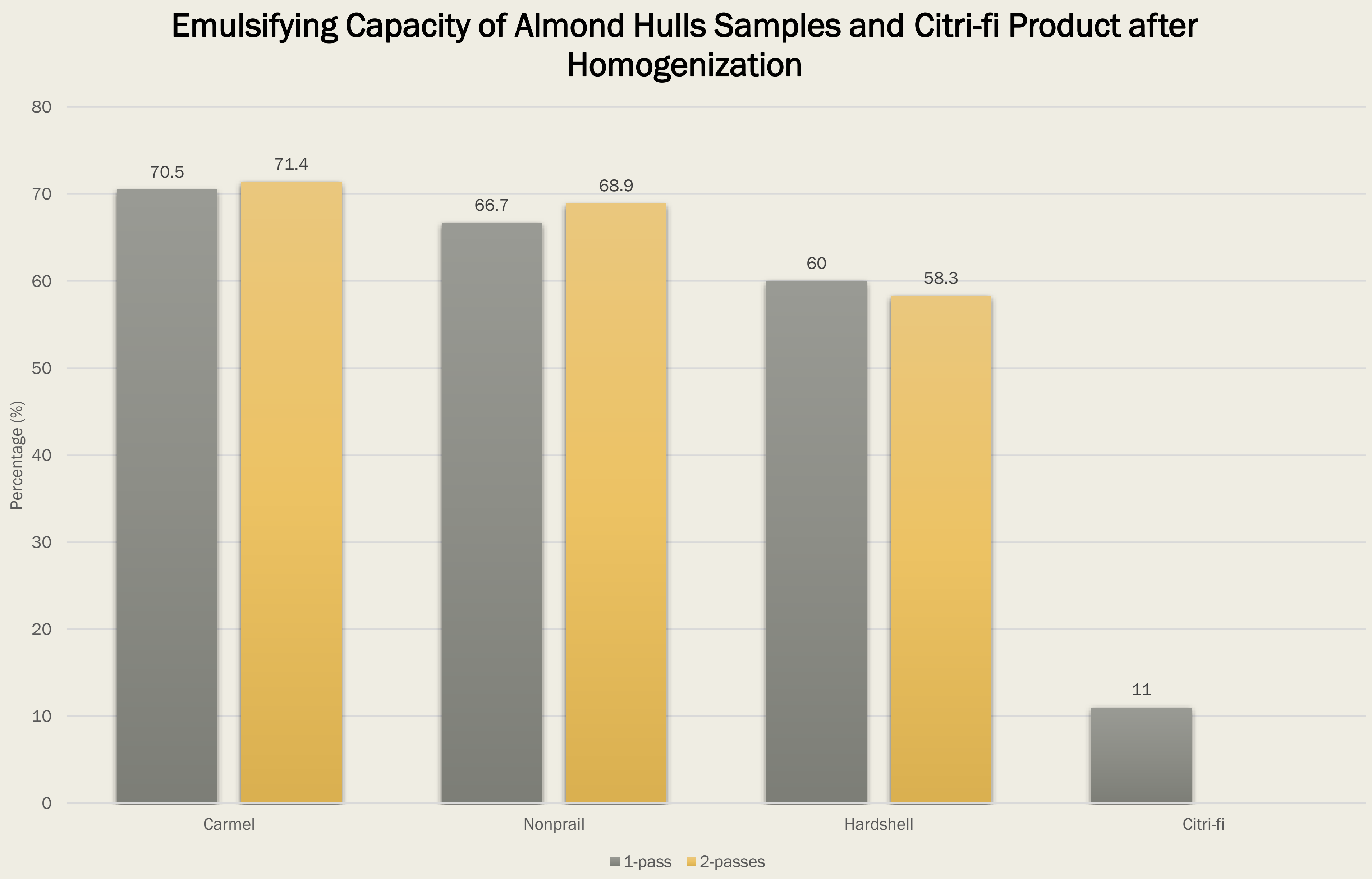


C0 – Untreated Carmel Samples
C1 – Pre-homogenized 1 pass Carmel Samples
C2 – Pre-homogenized 2 passes Carmel Samples

N0 – Untreated Nonpareil Samples
N1 – Pre-homogenized 1 pass Nonpareil Samples
N2 – Pre-homogenized 2 passes Nonpareil Samples
H0 – Untreated Hardshell Samples
H1 – Pre-homogenized 1 pass Hardshell Samples
H2 – Pre-homogenized 2 passes Hardshell Samples
Ci – Citri-fi 100® sample were obtained from Fiberstar company

Emulsifying Capacity (EC) Tested For the Almond hulls Powders After Homogenization

- (3% concentration) Add 0.75 g raw almond hulls powders produced by step 1 into 12.5 mL distilled water, vortex and add 12.5 mL oil. Dispersed with 10,000 rpm for 3min. Centrifuged with 500 g for 10 min, measure the height of each layer in the emulsifying system.
- EC = $\frac{height\ of\ emulsifying\ layer}{total\ height}$


Emulsifying Capacity of Almond Hulls Samples and Citri-fi Product after Homogenization
Percentage (%)
80
70
60
50
40
30
20
10
0
70.5
71.4
66.7
68.9
60
58.3
11
Carmel
Nonprail
Hardshell
Citri-fi
1-pass
2-passes

Summary and Preliminary Conclusions

- Preliminary results show that the almond hull samples contain unexpectedly high level of polyphenol contents especially the flavonoid contents and anti-oxidation capacity. Further process optimization is expected to release more anti-oxidants. Almond hulls could be a very good source of antioxidants that can be used in food and nutraceutical products.
- The enhanced oil binding capacity of almond hulls after homogenization was probably due to the increased porosity and surface properties of fibers.
- The promising level of oil binding capacity and emulsifying capacity indicates the potential use of the processed almond hulls as emulsifying agent, especially in water-in-oil food complexes to help stabilize high-fat products and emulsions.
- Previous literatures showed removal of phenolic compounds could resulted in improved emulsification and fat-binding properties (Pawar *et al*., 2001). This may suggest the extraction of antioxidants before using the dietary fibers in the meat products, beverage, bakery and other applications.

Next steps

- Pre-treat almond hulls suspensions with colloid mills to break down particle size and change internal structure to further increase porousity.
- Homogenize the samples with microfluidizer under different pressures and number of passes.
- Extract water soluble sugars and antioxidants before using the fibers as emulsifier and flavor carrier.
- Using HPLC and LC/MS to determine the compositions and contents of desired sugar and antioxidant compounds.
- Explore and develop different food and cosmetic applications based on the physiochemical properties of almond hulls.
- Optimize the processes and avoid chemical agents in the processes so that the processes are efficient and the products can be claimed “natural” and/or “clean label”.

Possible Almond Hall Applications

- High-performance ingredients
- Emulsifiers
- Concentrated syrup (Non-GMO)
- Extracted antioxidants as nutritional supplements
- Finished products
- Nutritional drinks
- Flavor-added jelly/paste
- Fermented almond hulls wine
- Anti-oxidant supplements in the form of tablets like grape seeds tablets

Possible Almond Hall Application Potentials

- Add value to the agricultural by-product
  - Making safe nutritious and lower cost foods
  - Increase bioactive compounds
  - All-natural, Non-GMO, clean label products
  - Multiple and flexible use
- Agricultural roots to solve real-world problem
  - Center for Biorefining has been focused on utilizing agricultural by-product and promote technology to transfer to industries
  - Studied citrus and many other fibers and successfully commercialized
  - Fast navigation from the lab to markets
- Natural products from ”feed to food directly”
  - Ranging from ingredients, food, beverage, supplements to personal care products
  - Require minimum additional processing from customers’ manufacturing processes

Acknowledgements

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- ABC provided the almond hull samples for the research
- We appreciate the contribution of researchers in Dr. Ruan's group to the experiment design, experiment execution, and collection and analysis of the data.
- We appreciate the discussion with Dr. Chen's group on analytical methodology.

Question?

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