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# Maximizing Oil Recovery From Corn Fermentation By-products

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# National ethanol production -

- 7.2 billion gal fuel grade in 2007
- 82% by dry-grind fermentation
- If 70% oil can be recovered, 440 million gal oil would be available for biodiesel production

# Composition of corn and fermentation by-products

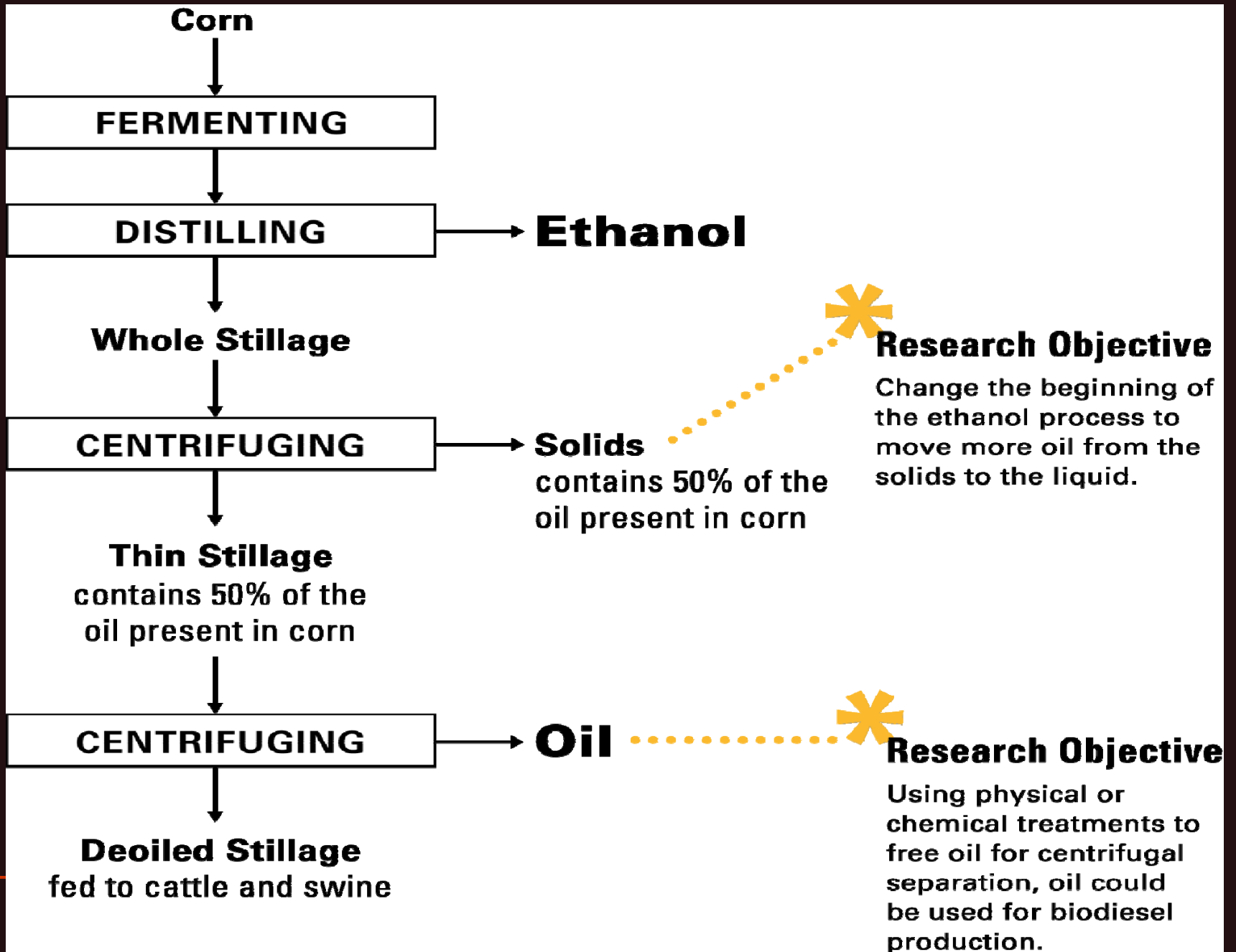
<b>Typical products</b>	<b>Oil, %</b>	<b>Protein, %</b>	<b>Carbohydrate + others, %</b>
<b>Corn</b>	<b>4</b>	<b>10</b>	<b>86</b>
<b>DDGS</b>	<b>13</b>	<b>31</b>	<b>56</b>
<b>Thick stillage or syrup</b>	<b>20</b>	<b>14</b>	<b>66</b>

## General approaches to extract oil

- Solvent extraction from DDGS (Janes et al, 2008 patent; Singh and Cheryan, 1998)
- Solvent extraction from liquid (UNL)
- Centrifugation of liquid after treatment (Winsness et al, 2007 patent)

## Our approaches to maximize oil extraction

- Upstream processing for oil release
- Downstream treatment to free the oil





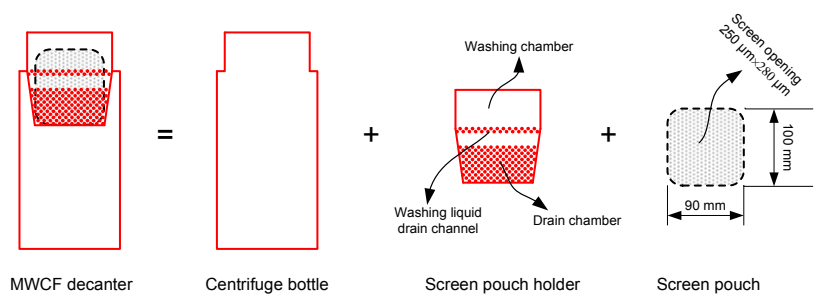
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# Effect of corn grinding method on oil partition to liquid phase

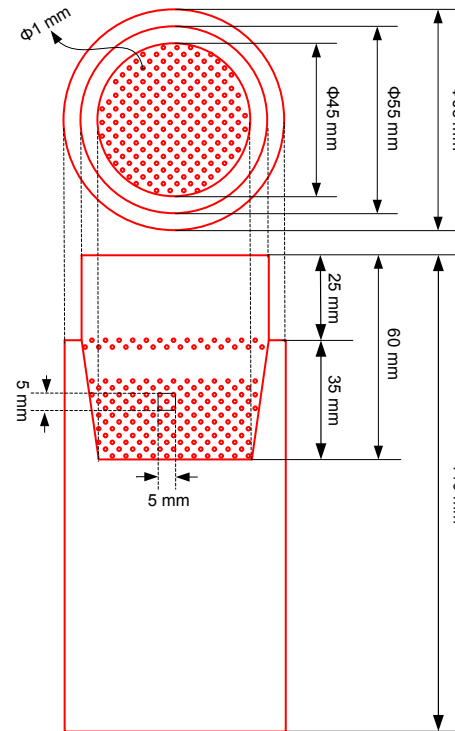
- Grinding to fine particle sizes
  - Extrusion to destroy cellular structure
  - Stillage separation by an unique MWCF process
  - Oil extraction and quantification
    - Free oil (FO) = Centrifugable oil
    - Trapped oil (TO) = Hexane-extractable oil
    - Bound oil (BO) = Non-hexane-extractable oil
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# Establishing a Multiple-Wash-Centrifuge-Filtration Device

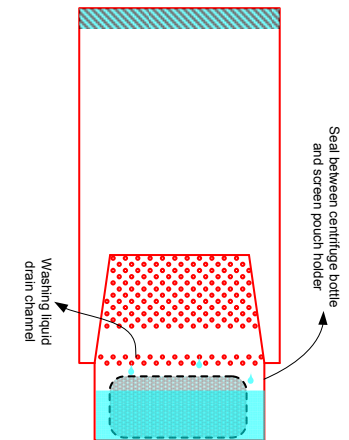
- Centrifugation and decanting work differently
- Lab simulation of industry process



A: assembly



B: construction

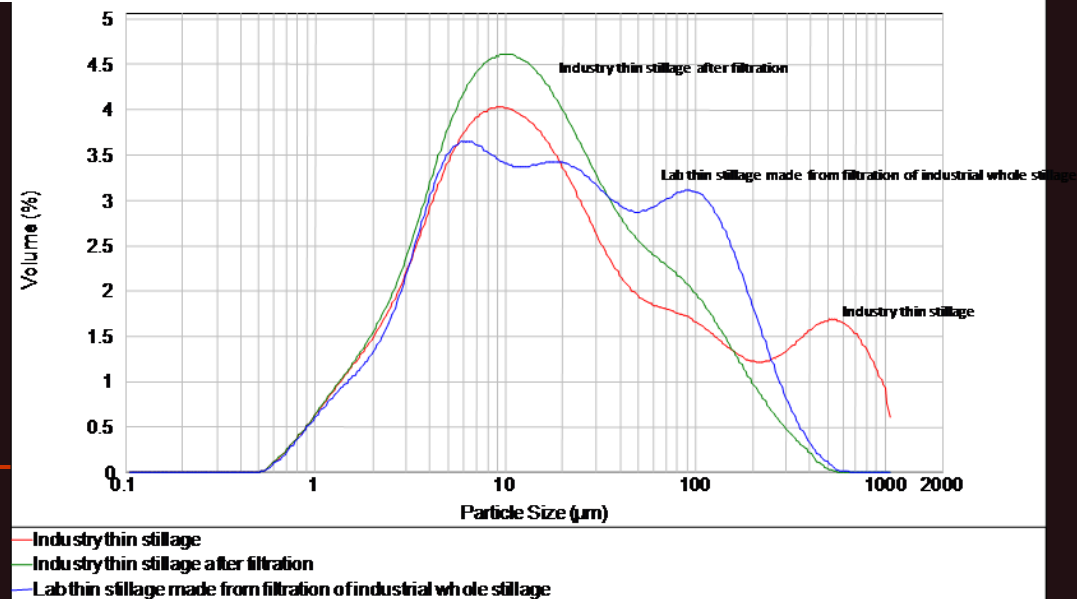
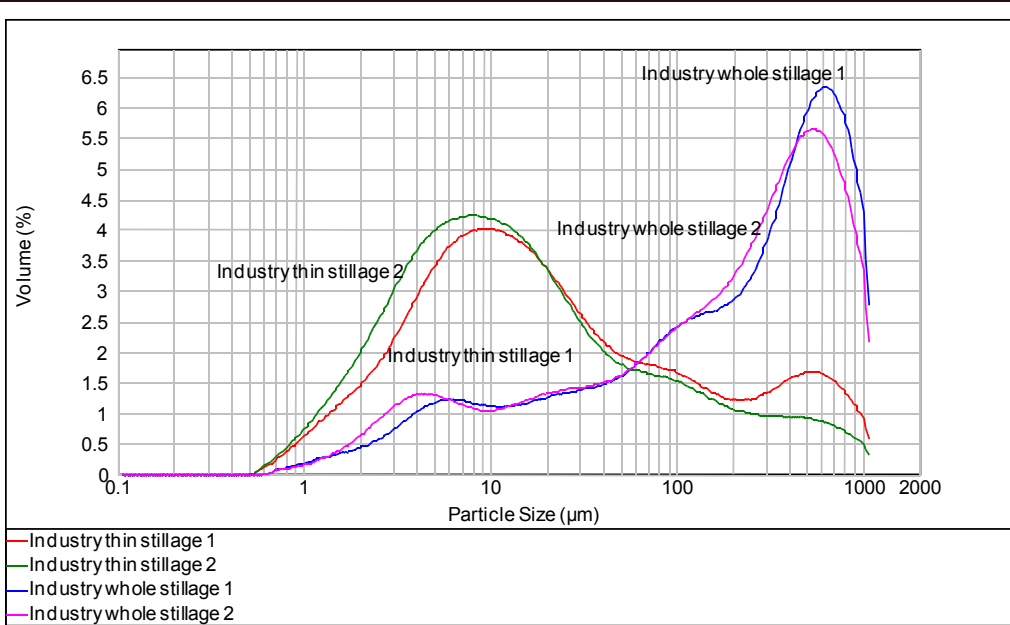


C: orientation during washing

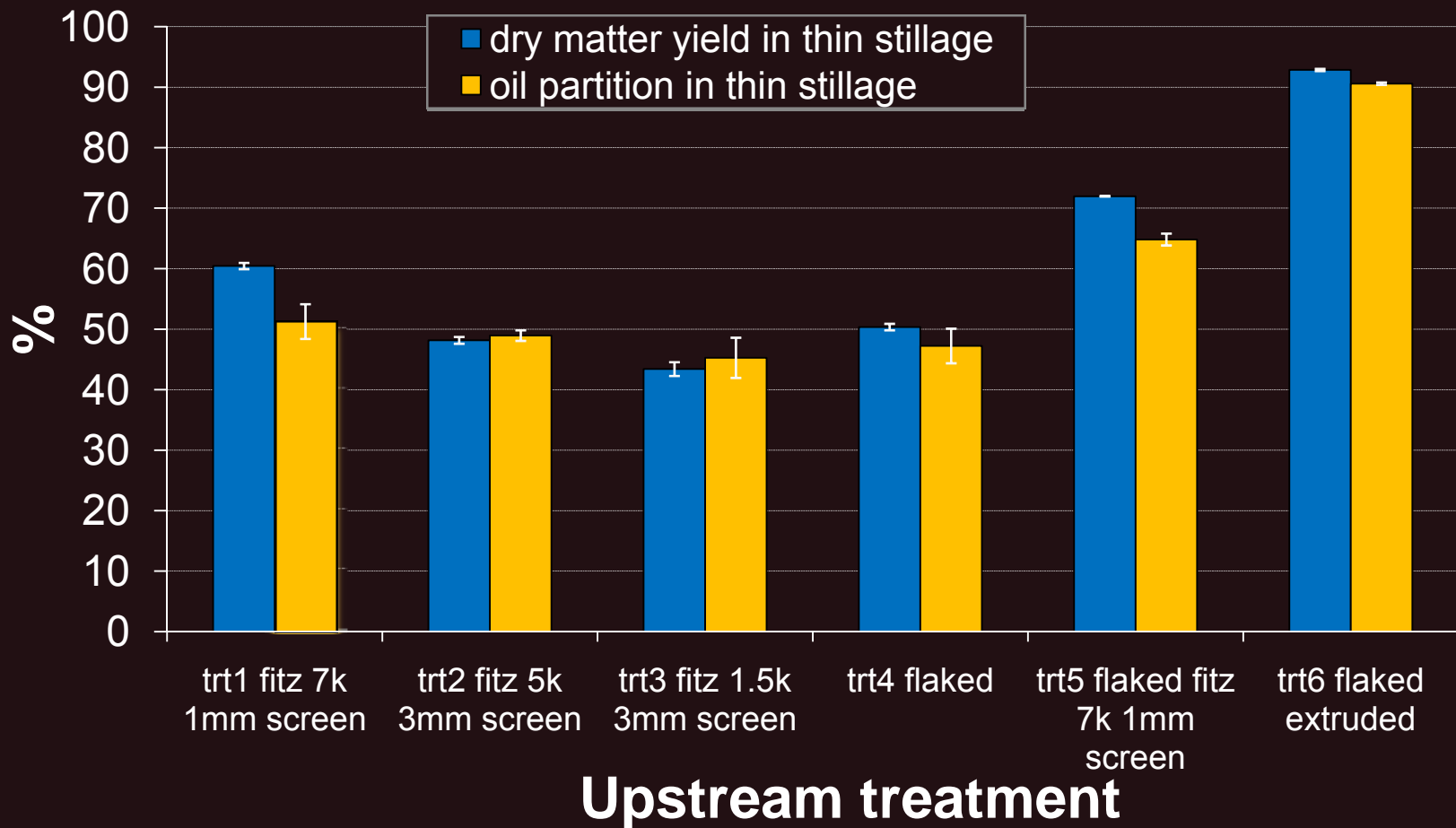
# Thin stillage composition by different treatment

	Solids, %	Wet matter yield, %	Dry matter yield, %
Industrial decanting	7.19 <sup>a</sup>	80.62 <sup>a</sup>	45.27 <sup>a</sup>
Lab centrifugation	5.08 <sup>b</sup>	59.31 <sup>b</sup>	23.17 <sup>b</sup>
Lab 4MWCF	7.31 <sup>a</sup>	83.26 <sup>a</sup>	48.35 <sup>a</sup>
LSD <sub>0.05</sub>	0.79	3.27	7.12

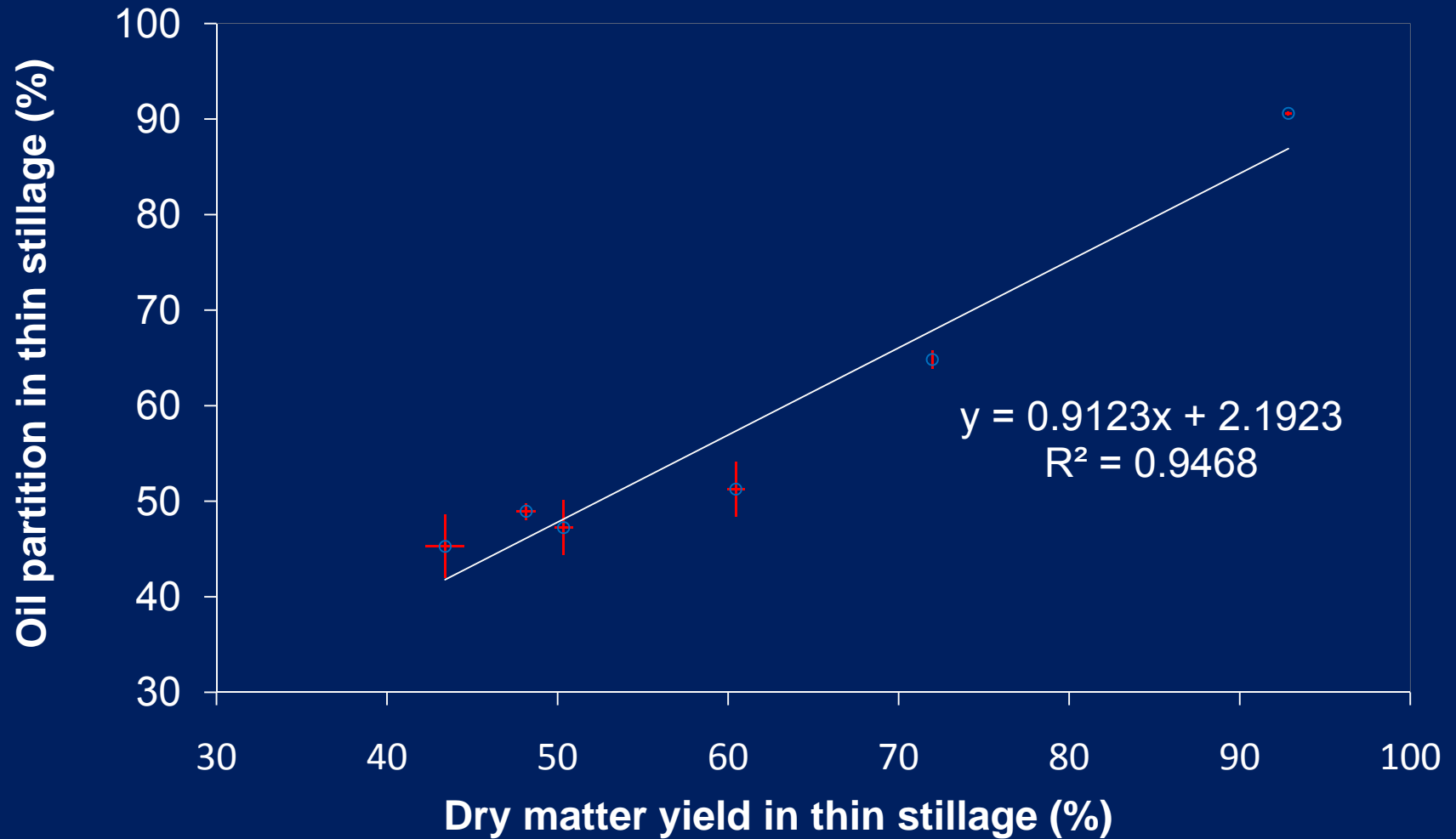
# Particle size distribution of stillages



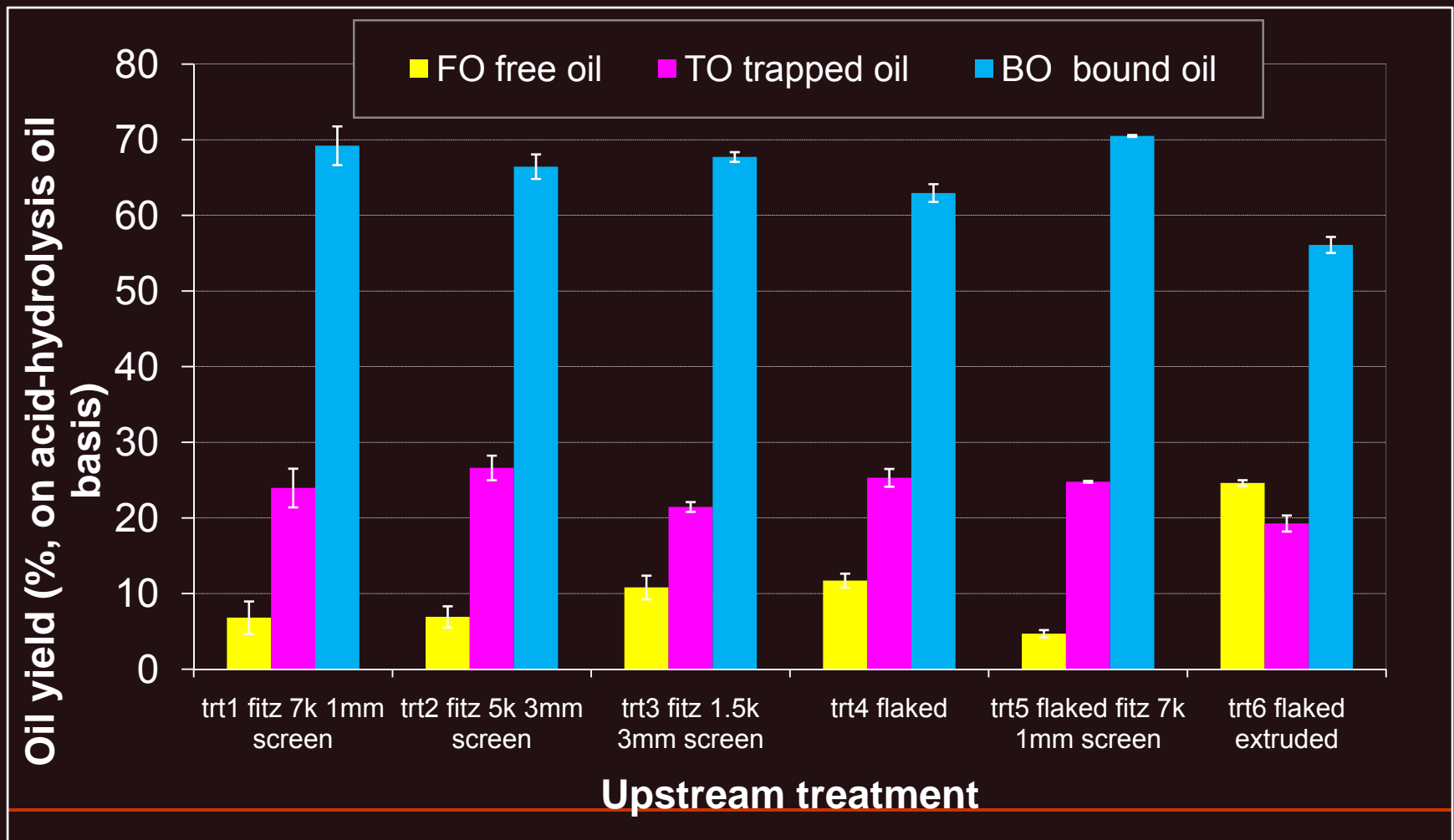
# Effect of corn grinding method on dry matter yield and oil partition into thin stillage



# Relationship between solid and oil distribution



# Effect of corn breaking method on the extractability of oil from fermentation mash



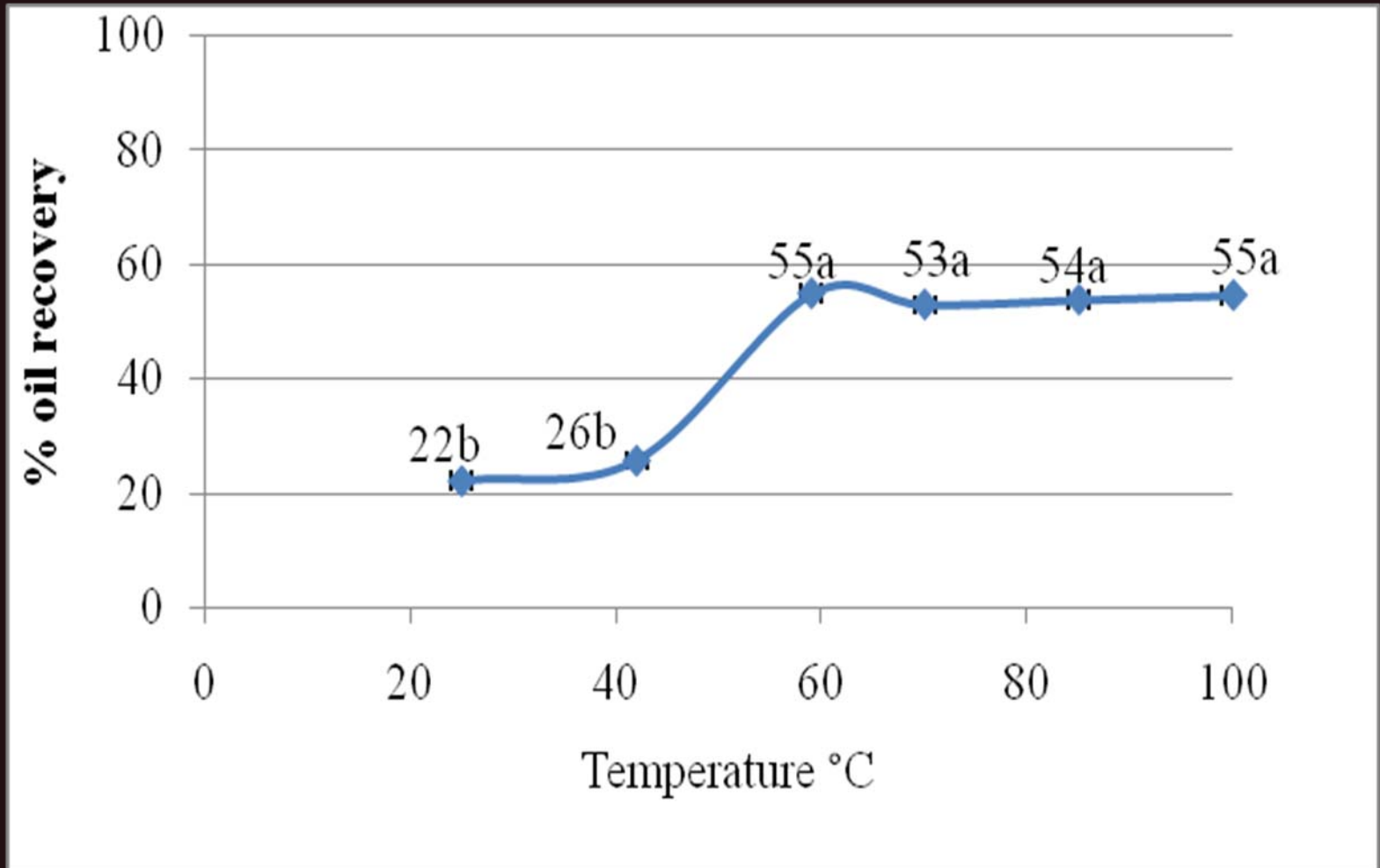


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# Oil recovery from the stillage

- Hypothesized forms of oil in stillage
    - O/W emulsion
    - Oil attached to hydrophobic surface
    - Oil bodies in coarse germ particles
  
  - Enzymatic treatment
    - Cellulases
    - Proteases
    - Hemicellulases
  
  - Physical treatment
    - Heating
    - Churning
-


# Oil recovery – Effect of temperature



# Effect of enzymes on oil recovery

% Enzyme	<sup>a</sup> Acid Protease	<sup>b</sup> Cellulases
	% Oil recovery	
<b>0</b>	64.9 ± 0.3d	60.1 ± 1.9abc
<b>1</b>	68.4 ± 0.4c	62.1 ± 0.4abc
<b>5</b>	69.3 ± 0.5cb	64.0 ± 0.3a ←
<b>10</b>	70.3 ± 0.7ab ←	63.2 ± 1.3ab
<b>15</b>	70.1 ± 0.5ab	57.9 ± 1.9c
<b>20</b>	70.5 ± 0.2a	59.1 ± 3.9bc

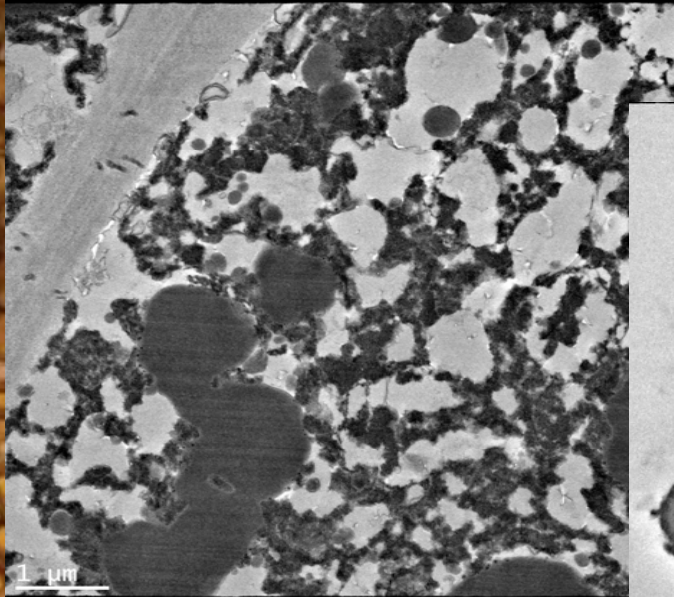
- <sup>a</sup> Protex™ 13FL from *Aspergillus niger*: pH 3.0, 50° C
- <sup>b</sup>Multifect® CX GC from *Trichoderma reesei*: pH 4.0, 55° C
- Means followed by different letters in the same column are significantly different



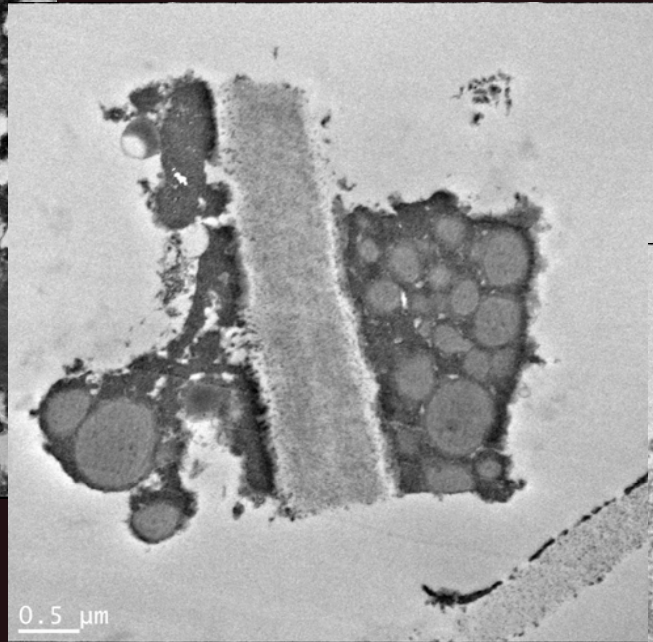
## Effect of particle size reduction on oil recovery

Enzyme treatment	% oil recovery	
	No Grinding	Grinding
A combination of enzymes	80.7 $\pm$ 1.0ab	82.6 $\pm$ 0.9a
No enzyme control	78.4 $\pm$ 2.4b	82.2 $\pm$ 1.4a

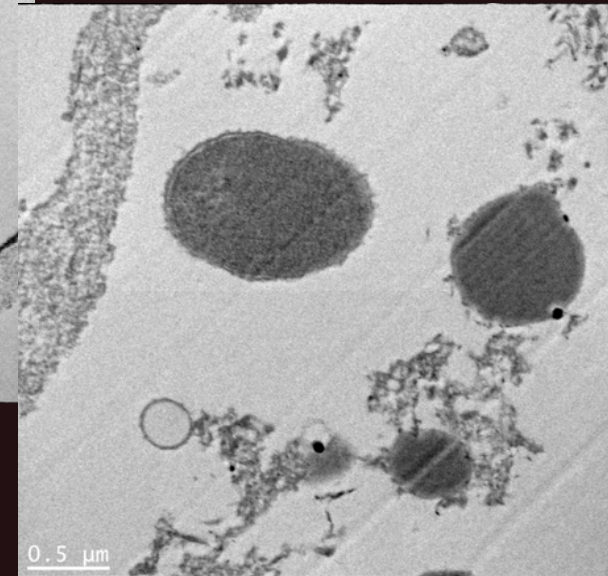
# TEM images of treated stillage samples



**Original stillage**



**Residue stillage after  
blending**



**Residue stillage after  
enzymatic hydrolysis**



# Effect of churning on oil recovery

Treatment	% Average oil recovery	
	3-hour incubation	6-hour incubation
Multifect <sup>®</sup> FE + Protex <sup>™</sup> 15L	78.6 ± 2.9	81.1 ± 14.8
No enzyme control	79.7 ± 1.9	75.0 ± 1.3



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# Conclusion

- Upstream proc
    - Cell disruption is needed for oil release, but it causes high meal fines in liquid
    - Oil partition in the liquid strongly correlates with solid content
  - Downstream proc
    - Enzyme hydrolysis increases oil recovery slightly
    - Heating treatment effectively improves oil recovery
    - Churning is effective in coalescing oil droplets
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# Acknowledgements

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