# Suga®Nate: A safer, milder, greener surfactant
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Recent Milestones:

- March 2007 – First use in a commercial shampoo in the US
- September 2009 – Approved for use in Australia
- December 2010 – Submitted to REACH for approval in EU
- October 2011 – Approved for use in Canada (DSL listing)
- June 2011 – USDA Certified Biobased Listed

This technology is eligible for the small business award. Annual sales for Colonial Chemical are less than $40 million.

Focus area:

- The design of greener chemicals

United States Component:
The technology described here has been developed and produced entirely within the United States. All of the work has been done at the facilities of Colonial Chemical, Inc., located in South Pittsburg, Tennessee.

Abstract
The unique, patented products described here represent a breakthrough in mild surfactant technology. They are produced from renewable resources, using naturally derived, biodegradable raw material. They are non-irritating to the eyes and skin, giving formulators of personal care products an opportunity to start with totally irritation-free ingredients.

These products are synthesized using raw materials that are nearly 90% renewable and could reach 100% as future development progresses. The synthetic pathway is very atom-economical; the only by-product is sodium chloride.

The reaction conditions used to make these products use only water as the solvent; the reaction conditions are very mild, especially when compared to the reaction conditions used for competing technologies, and there is no need for separation or purification steps.

The toxicity of these products is quite low in comparison to competing technologies and even lower than the starting raw materials! The products are also readily biodegradable and have no irritation to humans, both in eye irritation and skin irritation testing.

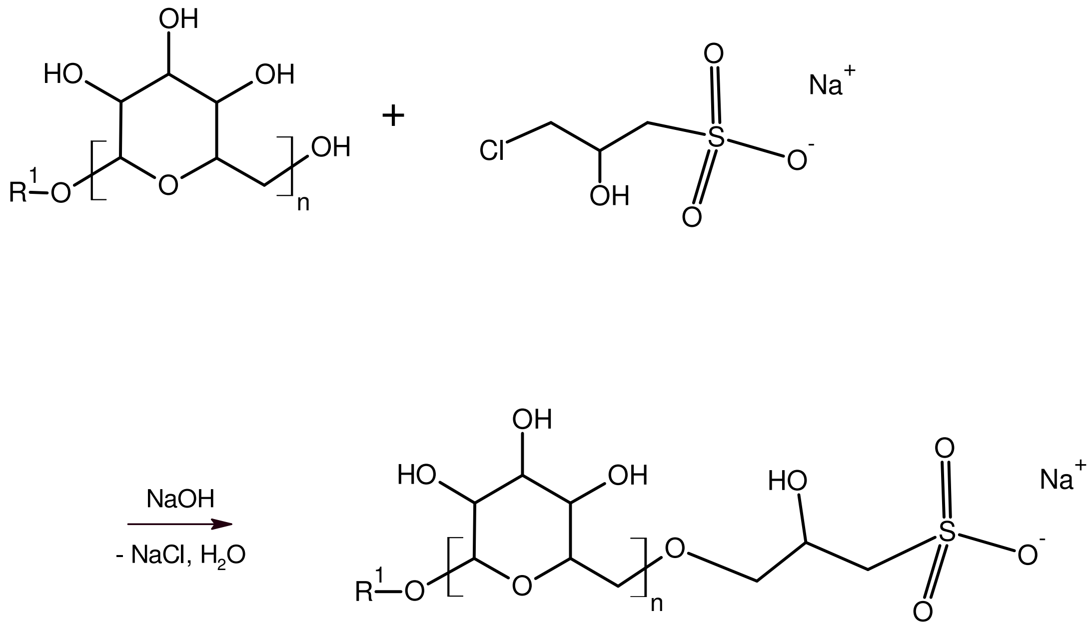
**Innovation and Scientific Merit**

New product synthesis
The new technology, sulfonated alkyl polyglucosides, was first invented at Colonial Chemical in 2002, and a patent was granted in September, 2003 (US 6,627,612). http://www.patents.com/us-6627612.html

The basic idea was to replace lauryl alcohol (much of which is produced from petroleum feedstock) as the hydrophobic component of surfactants with a natural, sustainable, and overall greener raw material.

Alkyl polyglucosides (APGs) have been commercial for many years, but are mainly used in the area of household and industrial cleaning. These products are made from 100% natural and renewable resources: corn sugar and fatty alcohols derived from coconut oil. However, the APG materials have some deficiencies that have prevented them from being widely used in personal care products, namely eye irritation and limited solubility. By introducing an anionic group to the alkyl polyglucosides, these deficiencies are overcome.

Our synthetic pathway is based on commercially available chemical intermediates that are widely used in the surfactant industry. Reaction of an APG molecule with the intermediate, sodium 3-chloro-2-hydroxypropyl sulfonate, is shown in the schematic below.




One unique feature of this chemistry is the specificity of the reaction. Although the reaction takes place in an aqueous solution that is slightly alkaline, there is no evidence that the 3-chloro-2-hydroxypropyl group undergoes hydrolysis to give a dihydroxypropyl sulfonate, and nearly all of the alkyl polyglucoside is reacted.[1]

The sulfonate intermediate is a well-known product of commerce that is used to produce sulfo-betaine surfactants (e.g., Cocamidopropyl Hydroxysultaine, http://www.colonialchem.com/Products/Personal-Care/Cola-Teric-Amphoterics/176/ColaTeric-CBS.

**Benefits to Health and the Environment**

Greener Chemicals

The chemical process to produce sulfonated alkyl polyglucosides is fundamentally different than most sulfonation reactions in the surfactant industry. The process to

[1] Analytical work performed by Technical Consultancy Services, June 4, 2009.

produce alpha-olefin sulfonate and sodium dodecylbenzene sulfonate utilizes $SO_3$ (sulfur trioxide) and air at high temperatures. Producing $SO_3$ involves burning elemental sulfur in air and can lead to release of 'acid-rain' pollutants.

On the other hand, the process to make sulfonated alkyl polyglucosides uses a chemical intermediate that is already widely used in the surfactant industry and is a comparatively safe chemical (CAS # 126-83-0). The reaction with alkyl polyglucoside is done at mild temperatures (80 – 90℃) and at a pH of about 9 – 10. The reaction is complete in about 4 hours, and the product is neutralized to pH of about 7. The only byproduct is NaCl.

Sulfate-type surfactants are also produced through a process that uses $SO_3$. These processes use high temperature conditions and have the potential to generate hazardous byproducts.

Currently, the two most common anionic surfactants used in shampoo, body wash and other personal care cleansing products are based on lauryl sulfate or the ethoxylated version, lauryl ether sulfate. A large percentage of the products are made synthetically from petroleum feedstock (ethylene).

Eye irritation and skin irritation of lauryl sulfate and lauryl ether sulfate products are recognized as high across the industry, and there are companies who will no longer use them in personal care formulations. 'Sulfate-free' has become a marketing label to differentiate these type of products.

A particular issue for ethoxylated lauryl ether sulfate is that ethoxylated alcohols that are used to make these products contain 1,4-dioxane at various levels. 1,4-Dioxane has been classified by the EPA as a probable human carcinogen:
http://www.epa.gov/chemfact/dioxa-sd.txt.
This contaminant causes concern with users and marketers of these products.

The technology described here, sulfonated alkyl polyglucosides, overcomes all of these problems. The molecule is made from renewable resources, and there is no eye irritation and no skin irritation. There is also no 1,4-dioxane present at any level in the product. Other benefits that these products bring include good biodegradability and very low toxicity to the environment.

The data in the tables below show the low level of toxicity, both in aquatic toxicity tests and in human irritation testing.

<u>Toxicity</u>

Table 1. Comparison of Aquatic Toxicity

| | Suga®Nate 160 | Sodium Lauryl Sulfate | Sodium Laureth Sulfate |
|---|---|---|---|
| Daphnia - 48 hour<br>ESA SOP 101<br>EC50 | 16.3 mg/L | 4.6 mg/L | 5 – 37 mg/L |
| Freshwater algae - 72 hour<br>USEPA Method 1003<br>IC50 | 52.9 mg/L | No data | > 65 mg/L |

| | | | |
|---|---|---|---|
| Fish larvae - 96 hour<br>ESA SOP 117<br>EC50 | 27.3 mg/L | 4.1 mg/L | > 8.9 mg/L |

Human irritation

EYE IRRITATION

HET-CAM: Hen's Egg Test Chorioallantoic Membrane

MatTek Epi-Ocular (In-vitro epidermal keratinocytes)

- Practically no ocular irritation potential in vivo, score of Zero
- Results indicate SugaNate 160 has a 'non-irritating' classification

HET-CAM SCORES

| Suga®Nate 160 | Sodium Lauryl Sulfate | Sodium Laureth Sulfate |
|---|---|---|
| 0.0 | 30.0 | 25.5 |

ACUTE SKIN IRRITATION

48 Hour Occlusive skin patch test on human volunteers

- No potential for dermal irritation

SKIN SENSITIZATION

Repeat Insult Patch testing (HRIPT)

- Does not indicate a potential for dermal irritation or allergic contact sensitization

Further data shown below shows that SugaNate 160 has an extremely good environmental profile, namely it is very biodegradable and does not have any genotoxic activity.

BIODEGRADABILITY

OECD 301 Ready Biodegradability Test (301E)

- 80-82% biodegradable

BACTERIA REVERSE MUTATION ASSAY

Ames test, OECD 471

- No detectable genotoxic activity at the non-cytotoxic concentrations associated with SugaNate 160, neither in the absence or in the presence of the S9 enzyme activation

Anti-microbial properties

An unexpected property of this new surfactant is the ability to withstand microbial contamination. We noticed that samples in our lab that were more than 4 years old did not show any propensity to be contaminated with microbial growth, even without the use of preservatives. This is contrary to the behavior of most surfactants that we encounter.

In order to quantify these observations, a sample was sent to an outside laboratory for microbial testing. The results in Table 2 are from material that was 2 years old when tested.

Table 2. SugaNate 160 Microbial Content

| Organism | cfu/gram |
|---|---|
| Aerobic Bacteria | < 10 |
| Yeast & Mold | < 10 |
| *C. Albicans* | Absent |
| *Staph. aureus* | Absent |
| Gram Neg. Bacteria | Absent |

These results confirmed that the 2-year old sample did not have any microbial content, but we wanted to test further whether the anionic surfactant would inhibit the growth of microbes when introduced to the material.

A second study was performed by the outside laboratory, this time using the 'Zone of Inhibition' test method. The results of this test are shown in Table 3, again using the 2-year old sample. The SugaNate 160 product was tested at both 16% and 8% active. A level of 16% is a normal range for a primary surfactant in higher-end shampoo formulations.

Table 3. Zone of Inhibition test results

| Sample | Psa | Ca | An | Total Score | Comments |
|---|---|---|---|---|---|
| 16% SugaNate 160 | 2 | 2 | 2 | 6 | Very good activity |
| 8% SugaNate 160 | 6 | 2 | 4 | 12 | Good Yeast & Mold; Poor *Pseudomonas* |

Organisms: Psa: *Pseudomonas aeruginosa* (gram negative bacteria)
Ca: *Candida albicans* (yeast)
An: *Aspergillus niger* (mold)
Scoring: 1 = Excellent; 2 = Very Good; 3 = Good; 4 = OK (moderate); 6 = Poor; 8 = No Activity

The conclusion, as stated by the outside testing expert is that the 'material can be considered to have reasonably good antimicrobial activity at the 16% active level. It would appear that using this material in formulations could contribute to enhancing overall resistance to microbial contamination'.

The advantage of this is that the level of antimicrobial additive used in formulations with this product can be reduced, and even in some cases eliminated.

## General Applicability of the Technology

The potential hurdles that must be overcome are few and seem to be manageable as evidenced by the increasing commercial use of the product. Foaming is not as voluminous as compared to the traditional lauryl sulfate and lauryl ether sulfate surfactants. The initial foam height of SugaNate 160 is slightly less than that of Sodium Lauryl Sulfate and Sodium Laureth Sulfate, as shown in Table 4 below. Although the original foam height is less, the foam height is actually maintained longer. The foam is a creamier consistency, and has smaller bubble structure compared to the foam generated from Sodium Lauryl Sulfate and Sodium Laureth Sulfate.

Table 4. Ross-Miles Foam Height

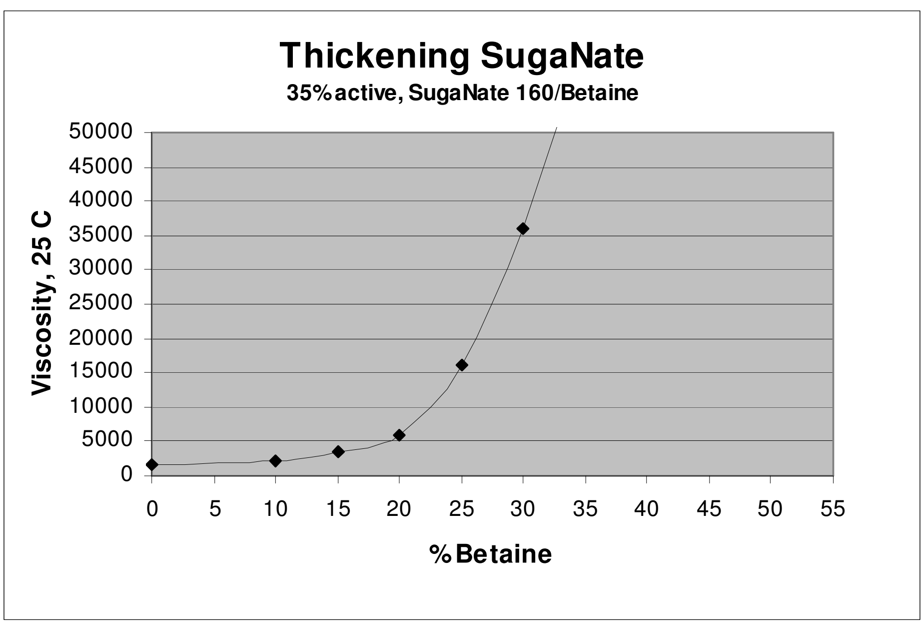
| **Ross-Miles, 1% solution** | **SugaNate 160** | **Sodium Lauryl Sulfate** | **Sodium Laureth Sulfate** |
|---|---|---|---|
| Immediate | 155 | 180 | 175 |
| 1 minute | 152 | 165 | 160 |
| 5 minutes | 150 | 160 | 155 |

Another challenge to formulating has been building sufficient viscosity in finished formulations. This challenge is a common feature of sulfate-free formulations With traditional sulfate formulations, a combination of sulfate, amide and/or betaine can be combined and will build viscosity with the simple addition of small amounts (< 5%) of sodium chloride.

The sulfonated alkyl polyglucosides do not build viscosity in response to addition of salt like traditional formulations. Thus, other ways of building viscosity into the formulations have to be found. Addition of naturally occurring polymers, e.g. gums and cellulosic polymers has been used by some to build viscosity. These methods are acceptable, but can have some drawbacks as well.

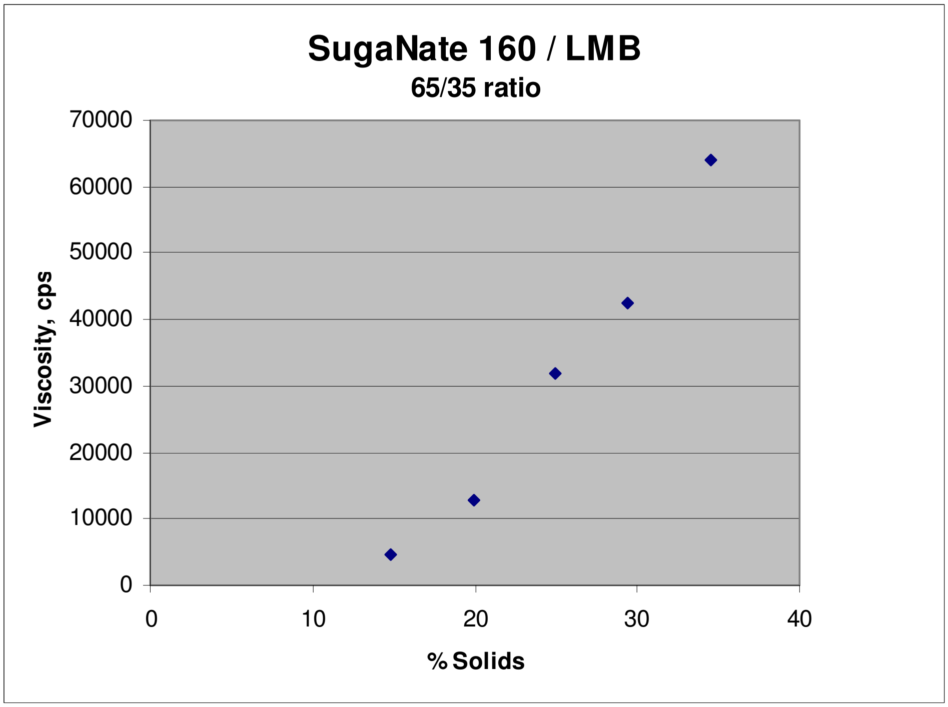
We have found that a simple combination of SugaNate 160 with a common amphoteric, lauramidopropyl betaine (LMB), will build viscosity close to 100,000 cps. A study on the effect of these two ingredients is shown in Figures 1 and 2 below. The first study was done at a fixed amount of 35% total surfactant in the solution. The relative amounts of SugaNate 160 and LMB were varied to give the curve shown in Figure 1.

Figure 1. Thickening SugaNate 160 with LMB



The second study was done at a fixed ratio of SugaNate 160/LMB (65/35) and varied the total amount of surfactant in the solution from 12% to 35%. It can be seen from this graph that high viscosity (> 30,000 cps) can be achieved at an active content of only 25%.

Figure 2. Viscosity vs. Total amount of Surfactant (SLHS/LMB)




## Conclusion

We have detailed here the design of greener surfactants; products that are unique, using patented technology.

- The products are produced from renewable resources,
- They are made using naturally derived, biodegradable raw materials,
- They are non-irritating to the eyes and skin,
- The reaction conditions are very mild,
- There is no need for separation or purification steps,
- The toxicity of these products is quite low,
- The products are readily biodegradable,
- They have comparable performance vs. competitive products.