Nature of Invention: Chemical molecule and synthesis route

**Applicant:** SynthoChem

**Inventors:** Pranjal Singh

**Chemical Formula: R(OCH2CH(OH)CH2)n(OGl)c where R = C8 to C10 alkyl chain and Gl = glucose unit**

**Chemical Name: Alkyl polyglucoside (AGP-0814)**

**Description:** Alkyl polyglucosides (APGs) are a type of surfactant that are commonly used in a wide range of personal care, household, and industrial applications due to their biodegradability, low toxicity, and good foaming and cleaning properties. AGP 0814 is more suitable for applications that require high solubility and foaming ability, such as personal care products and household cleaners.

**Chemical synthesis routes:**

a. **How can you prepare this chemical at lab-scale? List raw materials and chemicals needed. List reaction steps (product yield) and separation steps (final purity). If possible, give reaction conditions.**

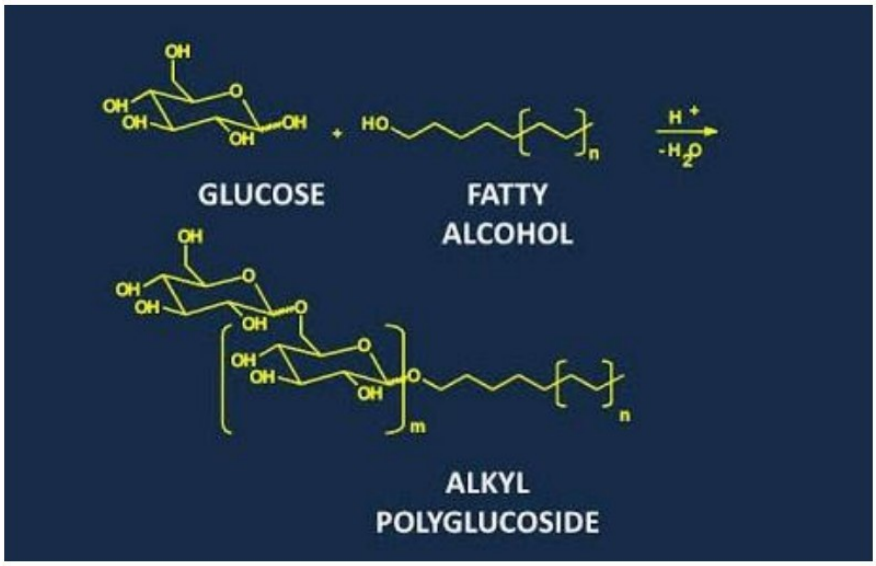
**Raw material:** Glucose, Fatty alcohol (C8-C10), HCl (for acid catalysis), water(solvent)

there are several preparative methods which lead to alkyl polyglycoside. As far as industrial production is concerned, Fisher's method is adopted successfully.

**Step 1: Preparation of raw material:**

Fatty alcohols are produced from natural oils and fats like coconut oil or palm kernel oil, whereas glucose is commonly produced from corn or potato starch.

**Step 2: Acid catalysed reaction of alcohol and glucose:**




**Procedure:**

Dissolve the glucose and fatty alcohol (e.g., Decanol/octanol) in water in the ratio of 1:2. Add a small amount of HCl (0.1M). Heat the reaction mixture under reflux conditions for several hours. (Optimal temperature 80-100 ®C). Once the reaction is complete, remove the reaction mixture from heat and allow it to cool to room temperature. Neutralize the reaction mixture with a suitable base (such as sodium hydroxide) to pH 7-8. Neutralized solution is pale yellow.

**Product yield = 50 % - 80 % (reported in research papers)**

**Step 3: Purification and separation:**

- Low boiling point by-products and unreacted reactants can be removed simply from heated reaction mixture.
- The excess fatty alcohol is removed by **vacuum distillation** and recycled to the reactor. This process can be repeated for 2-3 times to improve the purity. Residual fatty alcohol content in the product must be <1% because otherwise solubility and odour are adversely affected.

**Final purity = 99% (approximately)**

**Reaction condition: temperature = 80-100°C, pressure = 20-100 mbar**

**b. Are there any alternative synthesis routes to prepare this compound? List raw materials and chemicals needed. List reaction steps (product yield) and separation steps (final purity). If possible, give reaction conditions.**

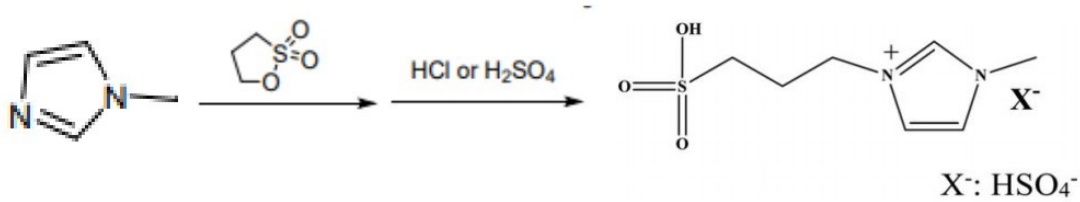
An effective process for synthesis of alkyl polyglycosides (APG) was developed using $SO_3H$-functionalized ionic liquid (SFIL) as catalyst. [PSmim][HSO4] shows the best catalytic performance among these four SFILs ([PSmim][$HSO_4$], [PSmim][*p*TSA], [PSPy][$HSO_4$] and [PSPy][*p*TSA]).

**Raw Material:** *N*-Methylimidazole, 1,3-propane sultone, toluene, ethyl acetate, copper (II) chloride, ethanol, sulfuric acid, glucose, n-Octanol

**Synthesis of SFIL:**

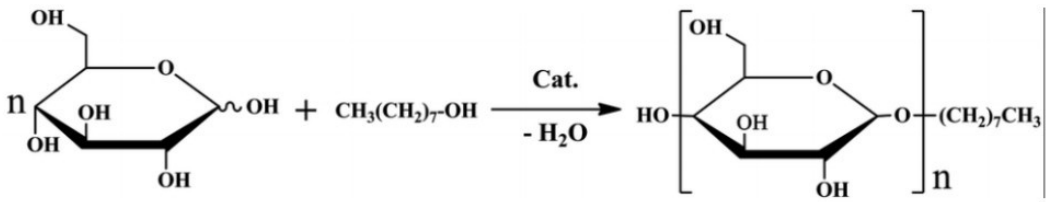
*N*-Methylimidazole (0.22 mol) and 1,3-propane sultone (0.22 mol) were dissolved in toluene (60 mL) as an inert solvent, which were added to a round-bottomed flask (200 mL) fitted with a reflux condenser and stirred at 343.15 K, 353.15 K, 363.15 K, 373.15 K for 24 h under a nitrogen atmosphere. Afterwards, the white precipitates were filtered off and washed several times with ethyl acetate to get 1-(3-sulfonic)-propyl-3-methyl imidazolium (PSmim), which was monitored by copper (II) chloride in ethanol and no blue colour could be found. Then, PSmim was dried in a vacuum under 343.15 K for 48 h. [PSmim][$HSO_4$] was obtained by a dropwise addition of one equivalent of concentrated sulfuric acid (98%) through constant pressure funnel to an aqueous

solution of PSmim that the mixture was stirred at 353.15 K for 12 h. Finally, the water of mixture was removed by a rotary evaporator, and then light yellow viscous [PSmim][$HSO_4$] was obtained. [PSmim][$HSO_4$] was dried under vacuum at 343.15 K for 48 h and stored in a desiccator.




**Glycosidation reaction:**

In the synthesis process of APG, a mixture of the glucose, *n*-octanol, and $SO_3H$-functionalized ILs were added to a round bottom flask (100 mL) fitted with a reflux condenser that was connected to Schlenk line and reacted at desired temperature for certain time. After the reaction finished, the mixture was cooled down to room temperature. Then, the yields of glycosides were determined by Fehling reagent.




**Reaction conditions: glucose (0.018 mol), *n*-octanol (5 equiv.), [PSmim][HSO4] (0.05 equiv.), reaction temperature (353.15 K), reaction time (24 h).**
**The conversion of glycose was measured using Fehling reagent = 92.8%**

**Separation process:**

When the glucosylation reaction finished, the product is divided into two phases after the addition of ethylacetate. The upper layer is a mixture of ethylacetate, excess *n*-octanol and APG, and [PSmim][$HSO_4$] and few glucose are present in the under layer. APG can be separated from the upper mixture by rectification and re-extraction. What's more, the [PSmim][$HSO_4$] of under layer can be reused without further treatment.

**%Purity = up to 95% (According to research paper)**

**References:**

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**List the contributions of each author:**

- Pranjal Singh did the complete R&D of the chemical including finding the chemical, synthesis routes, separation and % purity of the chemical.

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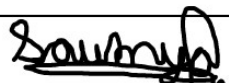
| Saumya Gupta (CEO) | 210944 |  |
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