NS207 Lab 3: Saponification and Esterification

Part I: Saponification (Soap Making)

Place 4ml olive oil into an evaporating dish. Stir in 3ml of 6M NaOH solution (YOU MUST **ABSOLUTELY** WEAR **EYE PROTECTION** IN THE LAB – THAT MEANS EVERYONE) and heat the mixture on a hot plate with constant stirring. When the mixture achieves the consistency of mayonnaise, remove it from heat and allow it to cool slowly.

Part II: Esterfication

Ester 1. Over the hot plate, boil some water in a beaker. In a test tube, mix 1ml 3M acetic acid, 1ml ethanol and 2-3 drops concentrated sulfuric acid. Heat the test tube in the bath until the contents of the test tube begin to boil – ama, do not let solution boil over. Once the solution has COOLED, add 2 ml deionized water, mix, and note the odor of the product. What does it smell like?

Ester 2. In a test tube, mix 0.5g salicylic acid and 1ml methanol. Stir well, and then cautiously add 3 drops concentrated sulfuric acid. Warm the solution gently for 5 minutes in a hot water bath, remove it and allow the mixture to cool to room temperature. Add 2ml deionized water, mix and note the odor of the product.
What does it smell like?

Ester 3. In a test tube, mix 1ml amyl alcohol (n-pentanol) and 1ml glacial acetic acid. Stir well, and then cautiously add 3 drops concentrated sulfuric acid. Warm the solution gently for 5 minutes in a hot water bath, remove it and allow the mixture to cool to room temperature. Add 2ml deionized water, mix and note the odor of the product.
What does it smell like?

Part III: Aspirin

Aspirin, or acetylsalicylic acid, is a very common ester. You can make Aspirin by treating salicylic acid with acetic anhydride. As acetic anhydride is **illegal** in Turkey, we will not be carrying out this experiment. However, here is an example of an acylation reaction:

Into a 125ml Erlenmeyer flask place 2g salicylic acid, 4ml acetic anhydride, and 3 drops of 85% (or concentrated) phosphoric acid. Stir the mixture well, and then heat the flask in a boiling water bath for 15 minutes. Remove the flask from the bath, and slowly add 2ml cold deionized water. Agitate the solution briskly for 1 minute, and then add 40ml cold deionized water. Agitate again and watch for the formation of white crystals. Cool the mixture in an ice bath for 15 minutes to allow for complete crystallization. Then collect the aspirin on a piece of filter paper using a vacuum filtration apparatus. Dry the material in the fume hoods.

$C_6H_4OHCO_2H + CH_3CO_2COCH_3 \xrightarrow{\text{phosphoric acid}} C_6H_4CO_2CH_3CO_2H + CH_3CO_2H$

Salicylic Acid Acetic Anhydride Acetylsalicylic Acid Acetic acid

Questions: Saponification

1. What is the mechanism of saponification? Please draw all steps and show the electron dance using arrows.
2. Do you think the reaction would have happened without heat? Why?
3. What is the effect of sodium hydroxide? Explain.

Questions: Esterification and Aspirin

1. Show the mechanism of ester formation for ester 1 and ester 3. Please draw all steps and show the electron dance using arrows.
2. Show the mechanism of ester formation in the case of aspirin (you can find the structure of aspirin from google, for example).
3. What is the limiting reactant in the case of the aspirin reaction?
4. Use stoichiometry to calculate the maximum theoretical yield for that reaction.

Hints: Calculate mass of acetic anhydride from its density, and then use stoichiometry to calculate number of moles participating in the reaction