Lab Activity 15: Aldol Condensation Reaction – Synthesis of Dibenzalpropanone
Adapted from "Monograph on Green Chemistry Laboratory Experiments", p. 13.

This aldol condensation reaction is "green" because hazardous organic solvents are avoided and reagents are non-toxic. **Answer these questions. DO NOT do this experiment.**

1. Preparation
   New structural features: the carbon adjacent to the carbonyl carbon is called an alpha (α) carbon (a different type of alpha carbon). The carbon next to the alpha carbon (two carbons away from the carbonyl carbon) is called a beta (β) carbon.
   a. In an aldol condensation reaction, the enolate ion reacts at the carbonyl carbon of an aldehyde or ketone in a nucleophilic addition reaction to form a _____ intermediate.
   b. The _____ intermediate accepts a proton to form a β–hydroxy aldehyde or ketone.
   c. The β–hydroxy aldehyde or ketone can undergo a _____ reaction to form a α,β–unsaturated aldehyde or ketone.
   d. Which reactant, benzaldehyde or acetone, has a H on an α carbon? State the approximate pKₐ of this acid.

2. Synthesis
   a. 3.8 ml (3.9 g) of benzaldehyde, 1.0 ml of acetone, and 15 ml of ethanol are added to a flask and shaken for two minutes.
      What is the function of ethanol?
      What is the mole ratio of benzaldehyde to acetone?
      Which reactant, benzaldehyde or acetone, is the limiting reactant?
   b. To this mixture, 30 ml of 10% NaOH is added and shaken for 10 minutes.
   c. The reaction mixture is cooled in ice and a pale yellow solid forms. This solid is _____ (also called dibenzylideneacetone or dibenzalacetone).

3. Workup
   a. Separate the pale yellow solid from the liquid by _____.
   b. Wash the pale yellow solid with ____.
      Look up the solubility of the pale yellow solid. Report the solubility of this solid in water and ethanol.
      (i) Solubility of _____ in water in mg/L.
      (ii) Solubility of _____ in ethanol in mg/L.
   c. Recrystallize the solid from _____. Would you use water or ethanol? Give reasons.
   d. Measure the yield.

4. Characterization
   a. The melting point of the pale yellow solid is 115-120°C.
      What is the true melting point of 4-hydroxy-4-phenyl-2-butanone?
      What is the true melting point of dibenzalpropanone?
   b. The IR spectrum of the pale yellow solid show peaks (in cm⁻¹) at 2900-3100, 1710, 1400-1600, 1350.
      Match each peak to a bond type.
   c. Is the product of this reaction 4-hydroxy-4-phenyl-2-butanone or dibenzalpropanone? Give reasons and numbers to support your answer.

5. a. A chemical reaction does not occur in Synthesis Step a. Why do you need to shake this mixture for 2 minutes?
   b. In Synthesis Step b, NaOH reacts with the acidic H in acetone in an acid-base reaction.
      Use curved arrows to show how NaOH reacts with acetone to form products.
      Does the equilibrium favor products or reactants? (Hint: compare pKₐ's of acetone to the conjugate acid of the base that reacts with acetone. You did this in Chem 12A.)
      If reactants are favored, how can you shift the reaction to the products side?
   c. Show the mechanism of this reaction. Use curved arrows to show how bonds break and form in each step.