

# STAO 2008 Session 2505 Two Chemical Demonstrations

## 3:30 – 4:30 pm, Friday, November 14, 2008 New York Room

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### Introduction

Two 10 minute demonstrations will be performed:

- (A) Sodium polyacrylate superabsorbent polymer (SAP)  
? gelling with water;  
? de-gelling with NaCl or vinegar.
- (B) Dyeing of polyamide fibres and fabrics with Kool-Aid  
(water soluble food colour dyes).

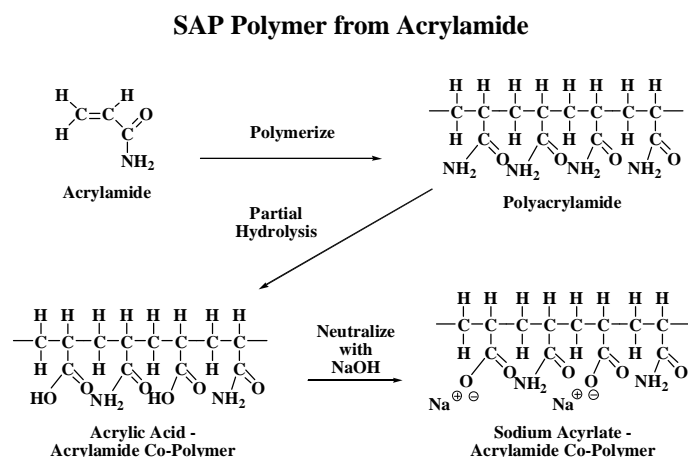
These two demonstrations are safe. They are suitable for students to perform in class. The materials used are not hazardous. The waste may be safely discarded down your sink and in your garbage.

### (A) Sodium polyacrylate superabsorbent polymer (SAP) ? gelling with water; ? de-gelling with NaCl or vinegar

#### Superabsorbent Polymers (SAP) of the Acrylic Acid Type

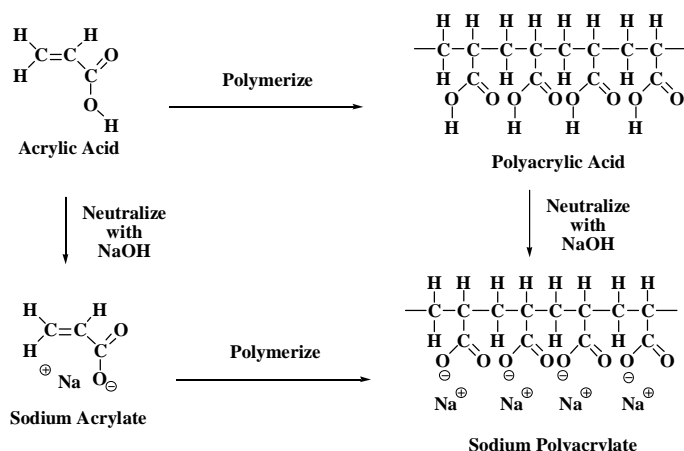
These polymers absorb water, forming gels containing 100s of times their mass of water. There are two general methods of synthesis for polyacrylic acid-type superabsorbents, either from **acrylamide** or from **acrylic acid** (1). The synthesis routes may be employed to produce either a non-cross-linked or a cross-linked polymer product.

Synthesis from acrylamide proceeds by polymerization, then partial hydrolysis of the polyacrylamide groups to create carboxylic acid side groups, and then neutralization of some or all of the carboxylate groups:



Synthesis from acrylic acid may proceed by polymerization followed by the neutralization of some or all of the carboxylic acid groups. Or the acrylic acid may be partially or completely neutralized and then polymerized:

### SAP Polymer from Acrylic Acid



#### Uses of SAP Polymers

SAP superabsorbent polymers were originally developed for agricultural uses. Diapers are the major use of the SAP polymers (90 %). Other uses are: incontinence products; sanitary pads; the horticultural industry; the food industry; specialty uses for water barriers in construction and electronics. Non-cross-linked sodium polyacrylate is used as a water softening agent in laundry deteratives.

#### Safe Handling of SAP Polymers

According to Buchholz and Graham (2) polymers made from acrylamide are unsafe for human contact, due to the difficulty of removing hazardous residues of the acrylamide, which is a carcinogen. If the method of synthesis of an SAP polymer is unknown then the substance should be considered as being potentially very hazardous.

#### Recovery of the SAP Polymer in Diapers

When a diaper is cut open, the first thing seen is a layer of what seems to be a fluffy padding. This is not the SAP, but is probably wood pulp fluff. Wood pulp fluff can absorb and hold roughly 10 times its mass of urine, and it gives the diaper its basic shape. The SAP polymer is found dispersed into the fluff, in the form of a white, granular solid. The SAP polymer can hold about 20 to 30 times its mass of urine.

There is an excellent reference article on the diaper polymer written by Mark Elliott of the company BASF (3). The article is available on the web and can be downloaded as a pdf file. It is 13 pages in length, well written, well illustrated, informative, and suitable for teachers or secondary students.

According to Mark Elliott, the diaper SAP absorbs water partly because of the water solubility of the ionic carboxylate salt of the polyacrylic acid groups, but mainly because of the osmotic movement of water into the polymer due to the sodium ions held inside the cross-linked polymer matrix. These factors cause the SAP to absorb water and form a gel, since the polymer molecules are too large to dissolve.

The gelled diaper polymer granules may be de-gelled by adding table salt, or by adding vinegar to the gel. Making the water salty reverses the osmotic movement of water into the polymer granules. Making the water acidic negates both the ionic solubility and the osmotic effect.

### SAP polymers: Exercises and demonstrations

#### Superabsorbent diaper polymer – collection and qualitative testing

Diaper polymer is a cross-linked sodium polyacrylate (SAP). The collection of diaper polymer granules, and the qualitative testing of the polymer granules has been described (4).

#### Materials and equipment required

Heavy Duty Diapers (e. g. Pampers Cruisers), scissors, large clear plastic bags, collecting vessels, newspaper or paper towels, table salt (NaCl); table vinegar (Acetic Acid, 5 % w / v); disposable vinyl gloves, small graduated cup or measuring vessel. The SAP polymer used in diapers is a cross-linked polymer made from sodium acrylate. It is non-volatile and is considered safe for human use, but the use of gloves is advisable when handling this material.

#### Instructions – diaper recovery and qualitative testing

The diaper polymer is dispersed as granules into wood pulp fluff, in separated pockets in the diaper. The polymer granules look like granulated sugar.

1. Wear disposable gloves. Spread out a large sheet of paper. Lay a diaper on the paper.
2. Cut along the rubbery part of the diaper on one side. You should be able to open out the inside part of the diaper, to see the layers of material inside. You want the layer that is fibrous. It resembles a cotton ball or a furnace filter material. It is in several pockets
3. Carefully place the cut-apart diaper into the plastic bag. The superabsorbent polymer granules are dispersed within the fibrous layer. Try not to lose any of the granules outside the bag.
4. From inside the bag, or from outside, shred the fibrous layer, and shake the bag. The polymer granules will fall out and fall to the bottom of the bag. Continue, until you get as much of the loose granules as possible.
5. Shake the granules down into one corner of the plastic bag. Put your collecting vessel on the sheet of paper. A beaker, a plastic cup, or a plastic weighing boat will be a good collecting vessel. If a balance is available, pre-weigh or tare the collecting vessel.

6. Cut off one bottom tip of the plastic bag with the scissors. Shake as much of the granules as possible into the collecting vessel. Re-weigh the vessel and the collected granules. Determine the mass of the collected granules. (As much as 12 g has been collected from a diaper.)
7. Divide the collected granules into 2 g portions.
8. Place one portion in a clean, clear cup or vessel. Add 50 mL of water. Stir or swirl. Observe the gelling behaviour of the granules in water.
9. Place another portion in a clean, clear cup or vessel. Add an equal volume of table salt. Add 50 mL of water. Stir or swirl. Observe the gelling behaviour of the granules in salt water.
10. Place another portion in a clean, clear cup or vessel. Add 50 mL of table vinegar. Stir or swirl. Observe the gelling behaviour of the granules in acidic water.

The polymer may be allowed to dry out for reuse or disposal. Dispose of all waste and excess polymer granules to the regular garbage.

#### Superabsorbent diaper polymer — quantitative measurement of water absorption

The diaper-type polymer material is subjected to many quality control tests. One of these is a centrifuge fluid retention test. The test fluid is physiological saline solution, sodium chloride solution, 0.9 % w / v. This test fluid is similar to urine in its absorption into the polymer. The industrial test uses heat sealable tea-bag paper, and a 1600 rpm centrifuge generating a 350 'g' force on the bag. This inspires a new respect for tea-bag paper.

Using the information from the literature search and some further information provided by Mark Elliott of BASF, the following exercise was developed. Recover diaper polymer granules as in the previous exercise. Mark Elliott suggested using a small clothes washer with a high speed spin. We use a small plastic kitchen salad spinner. This test requires a balance sensitive to **0.02 g**.

#### Quantitative salad spinner method for saline water retention ratio

1. Remove the staples from **four** staple-type flow-through tea bags. Remove the tea, the staples and the string.
2. Wear disposable gloves. Weigh out two samples of polymer granules, each of about **0.35 g**. Record the mass values of the polymer granules weighed out. Place each sample into an empty tea bag and staple shut securely with **one staple only**. Be sure you know which sample is in which bag!
3. Close and re-staple with only one staple two empty tea bags as controls

- Soak all four bags in **0.9 % w / v NaCl** solution for **20** minutes.
- Remove the bags from the saline solution, and arrange in the salad spinner to balance the mass. Spin as fast as possible for **one minute**.
- Pre-weigh a weighing boat or other vessel. Transfer each bag in turn to the vessel, and re-weigh. Determine the final mass of each bag.
- Calculate the average mass of the two empty control bags. Subtract the average control bag mass from the final mass of each of the filled bags. This gives the final mass of the polymer and retained fluid in each case.
- Subtract the mass of polymer from the mass of fluid plus polymer to determine the mass of retained fluid in each of the two test samples.
- Calculate the ratio of fluid retained to initial mass of the polymer sample in each of the two test samples. (Expected value of the ratio: about 20 to 30.)
- Report all observations, mass values and calculated results. Discard all waste into the garbage.

#### Soil-Moist™

Soil-Moist is an example of a horticultural superabsorbent polymer. It is available from Lee Valley Tools (5). Each 200 g jar (CAN \$12.50) contains 40 × 5 g packages of polymer granules. Other brands are also available.

The label on the container states that this is a polyacrylamide which **MUST NOT** be used for food plants. **Reminder:** this material should be considered hazardous. Use gloves and take appropriate care in handling the material.

#### Activity

Wear disposable gloves. When added to cold water (200 mL), one package of polymer granules will slowly swell to an extremely large volume (~ 200 mL?) of jelly-like material. The process requires several hours. It is faster with hot water. Adding salt or acid will reverse the absorption.

Allowing the polymer to slowly dry out will also reverse the process and the dry polymer granules will be recovered. Disposal may be into the solid waste (garbage).

#### Stiffy Stuff®

Stiffy Stuff is an example of a “fun” or magicians’ superabsorbent polymer. Stiffy Stuff is a sodium polyacrylate polymer. The material may be used to turn water to ‘slush’ or make it ‘disappear’. It is available from World Wonders (6). Other brands are also available (7).

It is pre-packaged into ~ 4 g portions in sealed paper packages, so it is very convenient to use. It costs \$ 0.25 per package in bulk. The SAP polymer in Stiffy-Stuff is a non-cross-linked sodium polyacrylate which World Wonders claims is non-hazardous. **Disposal may be into your sink.**

#### Activity

Wear disposable gloves. When added to room temperature water (100 mL), a quarter package of polymer powder (~ 1 g) will rapidly gel to an extremely large volume (~ 100 mL?) of slush-like material. The gelling is faster with hot water.

#### Tea bag demonstrations

Use flow-through type tea bags:

- Open the staple carefully without damaging the paper.
- Remove the staple, remove the tea powder, save the string and tag.
- Wear disposable gloves. **Carefully** fill the central portion of the bag with superabsorbent polymer granules (see caution below).
- Refold and close the bag.
- Re-staple, with the string held firmly by the new staple.

**Reminder:** these materials should be considered potentially hazardous. Disposal should be into the solid waste (garbage).

#### The iced-tea bag

Fill the tea bag with a quarter package of Stiffy Stuff. Use a cup of boiled water in a clear cup or container. When the “iced-tea” bag is added, the polymer will burst open the bag and the hot water will gel immediately. It will still be hot, but it looks like ice!

#### The incredible-bulk green-tea bag

Fill the tea bag with a package of Soil-Moist. Add a few drops of green food dye to the polymer for effect. Use a cup of boiled water in a clear cup or container. This is a slow-motion demonstration. The polymer granules will slowly swell. After an hour, it will probably burst the bag. In a day, it will fill the cup or vessel with a soft, green, jelly-like material.

#### (B) Dyeing of polyamide fibres and fabrics with Kool-Aid (water soluble food colour dyes)

There are eight food dyes considered safe and allowed for use in Canada. The eight allowed dyes are listed in the table below. They are produced in quantity for distribution in bulk to the food industry. You can see the colours of the aqueous solutions of the food dyes by visiting the website of a distributing company (8, 9).

Dyestuff	Dyestuff
Allura red or FD&C Red 40 (red)	Tartrazine or FD&C Yellow 5 (yellow)
Amaranth* or FD&C Red 2 (purple)	Fast green FCF or FD&C Green 3 (blue-green)
Erythrosine B or FD&C Red 3 (red)	Brilliant blue FCF or FD&C Blue 1 (blue)
Sunset yellow or FD&C Yellow 6 (red-yellow)	Indigo carmine or FD&C Blue 2 (blue)

\* Amaranth is allowed for use in Canada but not in the United States.

## Food colours from the grocery store

In Canada (Toronto), aqueous food dye solutions sold under the brand name Club House<sup>®</sup> and powdered drink mixes sold under the brand name Kool-Aid<sup>®</sup> are convenient sources of food dyes for dyeing fabrics

Club House brand food dye solutions are available in the four colours red, yellow, green, and blue. Neither the Club House bottle labels nor the Club House company website (10) have any information about which dyestuffs are contained in the solutions. However, this company is a division or subsidiary of the U. S. M<sup>c</sup>Cormick company: the dye contents of the corresponding M<sup>c</sup>Cormick brand solutions are available from the M<sup>c</sup>Cormick company website (11). It seems logical to presume that the same dyes are present in the Club House brand solutions.

Club House Food Colours / Presumed Dye Content	
Red	Allura red (Red 40) and Erythrosine B (Red 3)
Yellow	Tartrazine (Yellow 5) and Allura red (Red 40)
Green	Tartrazine (Yellow 5) and Brilliant blue (Blue 1)
Blue	Brilliant blue (Blue 1) and Allura red (Red 40)

Kool-Aid brand powdered drink mixes are available in a variety of formats, sizes, and flavours, with or without sugar and other sweeteners. The Canadian Kool-Aid labels have no information about which dyestuffs are contained in the powders but the dye contents of the corresponding products are available from the U.S. Kraft company website (12).

Kool-Aid Powdered Drink Flavours / Presumed Dye Content	
Cherry	Red 40 and Blue 1
Orange	Yellow 5 and Red 40
Lime	Yellow 5 and Blue 1
Grape	Red 40 and Blue 1

There is no information on any of these websites about dye amounts or concentrations in the various products.

## Dyeing fibres and fabrics

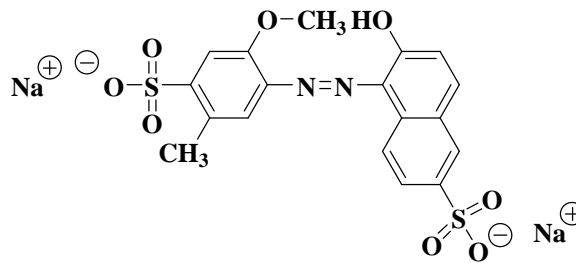
A dyestuff synthesis and fabric dyeing exercise is carried out in the organic chemistry laboratory of our three-year technology diploma program. Recently, Club House food colours and sweetener-free Kool-Aid powders were added to the dyes used in the experiment.

A part of the dyeing exercise is conducted using a multi-fiber ribbon, a material produced for the textile industry by the company TestFabrics Inc. (13). You can purchase lengths of the multi-fiber ribbon from Educational Innovations (14) for \$ 7.75 per yard, plus shipping. The 13 fibres on the ribbon are listed in the table.

The Multi-Fiber Ribbon		
Position	Fibre	Type
1	Acetate	cellulose, monoacetate
2	SEF*	acrylic
3	Arnel - bright	cellulose, triacetate
4	Bleached Cotton	natural cellulose
5	Creslan 61	acrylic
6	Dacron 54	polyester
7	Dacron 64	polyester
8	Nylon 6,6	polyamide
9	Orlon 75	acrylic
10	Spun Silk	natural polyamide
11	Polypropylene	polyolefin
12	Viscose	regenerated cellulose
13	Wool	natural polyamide

\* SEF is self-extinguishing fiber

The water-soluble food dyes belong to the class of dyes called acid dyes (1). Acid dyes are anionic in form, they are the water soluble salts of strong organic acids. These dyes remain in anionic form in strongly acidic solution. Allura red (1) may be taken as an example. It is an azo dye which is the disodium salt of the strongly acidic sulfonic acid groups.



**Allura Red (Red 40)**

Used in acidic solution, the anionic food colours are effective dyes for proteins and polyamides: natural hair; wool of all kinds, silks; nylons. Dyeing can be done in as short a time as 10 minutes, as noted in the instructions below.

There is a link in the Wikipedia article on Kool-Aid (1) to an article by Kristi Porter, in the magazine Knitty (15). This excellent short article gives you all the information needed to be able to dye natural hair, wool of all kinds, silks, and nylons with Kool-Aid or Club House food dye solutions.

Kool-Aid powders contain the required acid in the form of citric acid. Club House food dye solutions may be made acidic by adding some vinegar.

## Dyeing instructions

1. (Optional.) Soak the fabrics swatches or fibres to be dyed in a weak solution of detergent and wring dry.
2. Add the contents of one sugar-free package of Kool-Aid powder (makes 2.0 L of drink) to 200 mL of hot water (tap water is fine) in a large beaker. Cover with a watch glass if possible. Alternatively, use 5 mL of a Club House food dye solution, add 25 mL of vinegar, and dilute with tap water to 200 mL.
3. Add the prepared fabrics. Stir gently and keep the solution as hot as possible for at least 10 minutes (longer is better).
4. Place the beaker in a sink (caution: hot) and run in cold tap water until the rinse water runs clear and colourless, taking care not to rinse the fabric swatches down the drain.
5. Hang the fabrics on a line to dry. Repeat the rinsing and drying if it is intended to keep the fabrics.

## Forensic testing for wool, nylon, and silk

The Kool-Aid dyeing process can be used to determine whether or not an unknown fabric or fibre sample is a polyamide (wool, nylon, or silk). If a fibre labeled as wool or silk, or a rope labeled as nylon does not absorb dye, it may very well not be what it claims to be.

## Other extensions

Ask students to supply other samples for dyeing, such as animal hair, unusual wools, insect cocoons, spider silk, Kevlar<sup>®</sup>, Nomex<sup>®</sup>, etc.

## Acknowledgements

The author wishes to thank the department technologists, Bill Rolfe, Fiona Anthony, and Randy Travis for their invaluable and cheerful assistance in the development of the demonstrations performed at this session. Thanks are due to Randy Travis for taking the photographs.

## Contact Information

Professor Cash will retire at the end of February, 2009. Inquiries about this material may thereafter be addressed to him at: [dn.cash@uclmail.net](mailto:dn.cash@uclmail.net)

To arrange a working visit to Mohawk College contact Professor Cindy Mehlenbacher. If you have questions about the methods, contact Professor Dan Wilson (dyeing) or Technologist Randy Travis (SAP). To request materials (see page 6), contact Chief Technologist Bill Rolfe.

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A pdf file version of this handout may be downloaded from:

<http://uclmail.net/users/dn.cash>

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## Materials Package

### Compliments of Mohawk College

#### Chemical, Environmental, and Biotechnology Department

1. Diaper SAP<sup>1</sup> (5 g)  
? harvested from Pampers Cruisers
2. Soil Moist SAP<sup>2</sup> (5 g)  
? purchased from Lee Valley Tools
3. Stiffy Stuff SAP<sup>3</sup> (4 g)  
? purchased from World Wonders
4. Multi-Fiber Ribbon<sup>4,5</sup> MFF #43 (20 cm)  
? purchased from TestFabrics Inc.

#### Contacts

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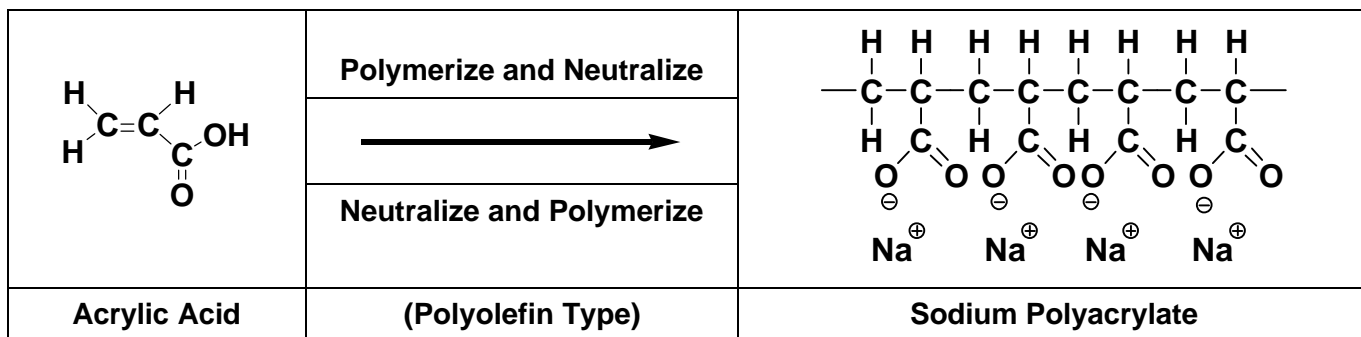
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[randy.travis@mohawkcollege.ca](mailto:randy.travis@mohawkcollege.ca)

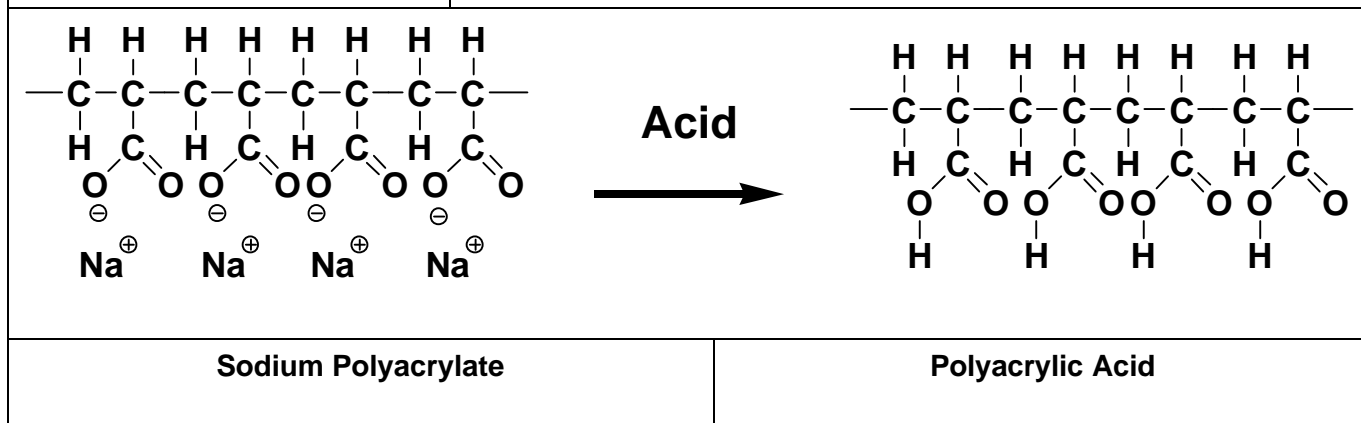
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## Superabsorbent Polymer (SAP) – Sodium Polyacrylate Type

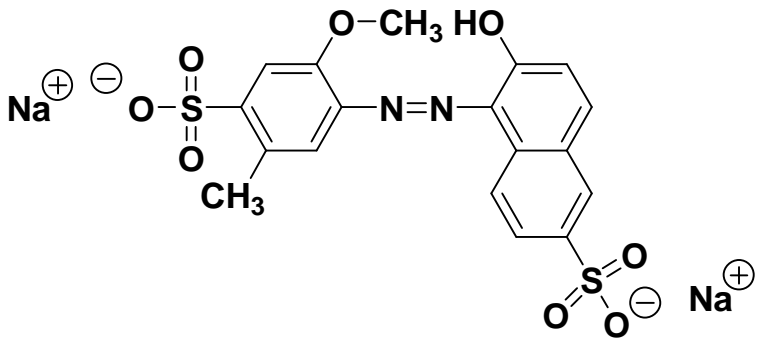


<b><u>Gelling Behaviour</u></b>	? superabsorbent gelling with water (100s to 1) ? ionic and osmotic forces attract water to the polymer ? polymer is water soluble and non-hazardous
<b><u>De-Gelling Occurs If</u></b>	? water is highly osmotic ? water is acidic ? polymer gel is squeezed like a sponge



<b><u>Commercial Applications of Cross-linked Polymers</u></b>	? diapers, incontinence pads, sanitary pads, packaging ? horticultural polymers ? engineering (waterproof joints and seals)
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## Dyeing with Water Soluble Food Colour Dyes

<p><b><u>Acid Dyes</u></b></p> <p>? water soluble salts of strong organic acids</p> <p>? effective dyes for polyamides: wool, hair, silk, nylons</p>	 <p style="text-align: center;"><b>Allura Red (FD&amp;C Red 40)</b></p>
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<p><b><u>Food Colour Dyes</u></b></p> <p><b><u>in Kool-Aid</u></b></p> <p>? three dyes only</p> <p>? Red 40, Yellow 5, Blue 1</p>	<table border="1" style="width: 100%; border-collapse: collapse; text-align: center;"> <tr> <th colspan="2">Kool-Aid Powdered Drink Flavours / Dye Content</th> </tr> <tr> <td style="width: 50%;">Cherry</td> <td style="width: 50%;">Red 40 and Blue 1</td> </tr> <tr> <td>Orange</td> <td>Yellow 5 and Red 40</td> </tr> <tr> <td>Lime</td> <td>Yellow 5 and Blue 1</td> </tr> <tr> <td>Grape</td> <td>Red 40 and Blue 1</td> </tr> </table>	Kool-Aid Powdered Drink Flavours / Dye Content		Cherry	Red 40 and Blue 1	Orange	Yellow 5 and Red 40	Lime	Yellow 5 and Blue 1	Grape	Red 40 and Blue 1
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Cherry	Red 40 and Blue 1										
Orange	Yellow 5 and Red 40										
Lime	Yellow 5 and Blue 1										
Grape	Red 40 and Blue 1										

The Multi-Fiber Ribbon		
Position	Fibre	Type
1	Acetate	cellulose, monoacetate
2	SEF	acrylic
3	Arnel - bright	cellulose, triacetate
4	Bleached Cotton	natural cellulose
5	Creslan 61	acrylic
6	Dacron 54	polyester
7	Dacron 64	polyester
8	Nylon 6,6	polyamide
9	Orlon 75	acrylic
10	Spun Silk	natural polyamide
11	Polypropylene	polyolefin
12	Viscose	regenerated cellulose
13	Wool	natural polyamide



## Families of Acrylic Polymers

	<b>Acrylic Acid</b>	<b>When polymerized, give rise to various types of superabsorbent materials (SAP)</b>
	<b>Acrylamide</b>	
	<b>Acrylonitrile</b>	<b>When polymerized, gives rise to acrylic fibres (e. g. Orlon<sup>®</sup>)</b>
	<b>Methyl Methacrylate</b>	<b>When polymerized, gives rise to acrylic glasses (e. g. Plexiglas<sup>®</sup>)</b>
	<b><math>\alpha</math>-Cyano Methacrylate</b>	<b>When polymerized, gives rise to superglue adhesives</b>

## Materials Package

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1.	<b>Diaper SAP (5 g)</b> <b>? harvested from Pampers Cruisers</b>
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3.	<b>Stiffy Stuff SAP (4 g)</b> <b>? purchased from World Wonders</b>
4.	<b>Multi-Fiber Ribbon MFF #43 (20 cm)</b> <b>? purchased from TestFabrics Inc.</b>


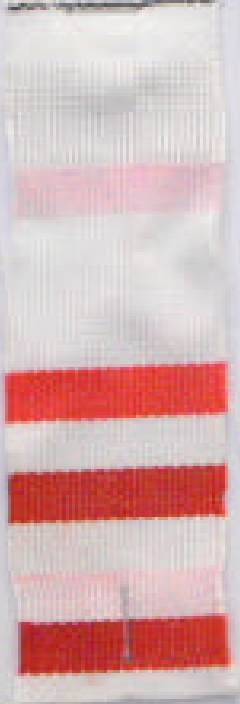









**See the printed handout for more information  
and contact details.**

**The handout and overheads are available for download  
as a pdf file version at <http://uclmail.net/users/dn.cash>**

## Dyeing with the Multi-Fiber Ribbon



## Dyeing Fabrics with Kool-Aid

Dyeing Fabrics with Kool-Aid®			
Undyed Control	Cherry Kool-Aid	Multi-Fiber Ribbon	Grape Kool-Aid
		<ul style="list-style-type: none"> <li>— Acetate —</li> <li>— SEF —</li> <li>— Arnel - Irlight —</li> <li>— Bleached Cotton —</li> <li>— Cordan 61 —</li> <li>— Dacron 54 —</li> <li>— Dacron 64 —</li> <li>— Nylon 6,6 —</li> <li>— Orlon 75 —</li> <li>— Spun Silk —</li> <li>— Polypropylene —</li> <li>— Viscose —</li> <li>— Wool —</li> </ul>	
		Silk Fabric	
		Nylon Fabric	
		Needlepoint Wool	
		Nylon Rope	