# CHAPTER V

## CONCLUSION and SUGGESTION

The research on synthesis and characterization of modified acrylamide-pyrrolidone copolymer under the various dose rates and total doses, several attempts were used, based on theoretical considerations, to pursue the possible technologies to synthesize polyacrylamide by gamma irradiation, hydrolysis of polyacrylamide and copolymerize partially hydrolyzed polyacrylamide(HPAM) with 1-vinyl-2-pyrrolidone by gamma irradiation for increasing the water absorption. At the stage, one can conclude that a random terpolymer of polyacrylamide-polyacrylate-poly(vinyl pyrrolidone) was obtained as a highly-temperature resistance absorbence. Both the positive and negative effects to the above mentioned purposes are found. The positive influences toward enhancement of the conversion of monomer and water absorption are, of course, the driving force for a scale-up production. The negative part of the result is the master for future improvement. However, both are presented as the following.

1. Polyacrylamide was synthesized and confirmed by FTIR spectroscopy. The absorption bands at 3254, 3196 and 1414 -1662 $cm^{-1}$ confirmed the characteristics of the $-CONH_2$ group. The C-H stretching of $CH_2$ band, which indicated the backbone of the homopolymer containing the $-CH_2-$ group appeared at 2940 and 1452 $cm^{-1}$.

2. Partially hydrolyzed polyacrylamide was synthesized by hydrolysis of polyacrylamide with NaOH and degree of hydrolysis by semi-micro Kjeldahl method was determined and confirmed by FTIR spectroscopy. The stretching absorption peak of the $-CONH_2$ group shows weaker bands in the 3175-3227 $cm^{-1}$ region(corresponding to the $-NH_2$ group) than the previous

polyacrylamide spectra, the absorption peaks of the -COO group appeared at 1564, 1407 $cm^{-1}$ and the assignment of the band in the 3227 $cm^{-1}$ region was the bound water.

3. Partially hydrolyzed polyacrylamide(HPAM) and 1-vinyl-2-pyrrolidone were copolymerized by gamma irradiation and confirmed by FTIR. The FTIR absorption of partially hydrolyzed poly(acrylamide-co-1-vinyl-2-pyrrolidone) demonstrated characteristic bands for both partially hydrolyzed polyacrylamide and poly(1-vinyl-2-pyrrolidone). The assignment of the band in the 3300 $cm^{-1}$ region to bound water was possibly made according to the similar absorption in the spectrum of partially hydrolyzed polyacrylamide. Other bands include C-H stretch vibration at 2942 $cm^{-1}$. Carbonyl amide band occurs as a strong peak near 1664 $cm^{-1}$ close to the frequency observed in the specturm of poly(1-vinyl-2-pyrrolidone). Methylene deformation peaks in the 1323-1444 $cm^{-1}$ region have the same pattern as in the spectrum of poly(1-vinyl-2-pyrrolidone). The band at 1291 $cm^{-1}$ represents the C-N stretching mode of 1-vinyl-2-pyrrolidone monomers units.

4. By increasing the quantities of total dose increases the percent conversion of the monomer. The highest conversion of polyacrylamide occured at the total irradiation dose of 1.4 kGy. Higher total dose decrease the percent conversion slowly. Water absorption capacity of partially hydrolyzed poly(acrylamide-co-1-vinyl-2-pyrrolidone) was in the range of 730 to 950 times their original dried weight depending upon the quantities of total dose, based on a 1:2 ratio of partially hydrolyzed polyacrylamide (g) and 1-vinyl-2-pyrrolidone (g). The highest conversion of monomer and water absorption of copolymers occured at the total irradiation dose of 10 kGy.

5. Increasing the quantities of dose rate increases the percent conversion of the monomer. At dose rate of $1.19 \times 10^4$ Gy/h percent conversion of the monomer is highest, so is the water absorption value of copolymers.

6. The highest water absorption occured at the amount of HPAM 5 g. The higher quantity of HPAM gives the lower absorption.

7. The highest water absorption occured at the quantity of 1-vinyl-2-pyrrolidone give the lower water absorption, likewise, the quantity of 1-vinyl-2-pyrrolidone lower than 0.94 M also give the lesser water absorption.

8. Water absorption capacity of partially hydrolyzed polyacrylamide increased with increasing the degree of hydrolysis up to 71%. When the degree of hydrolysis is higher than 71% the water absorption is constant. The water absorption of partially hydrolyzed poly(acrylamide-co-1-vinyl-2-pyrrolidone) is highest at the degree of hydrolysis of 71%. Beyond this value, water absorption decreased.

9. Drying method has a strong influence on water absorption. Heat (oven) drying method gives the product with less water absorption than does the freeze drying method. The water absorption capacity of the freeze dried method was 1,100 g/g while that of the oven dried method was only 950 g/g times their original dried weight. Heat drying induces the crosslinking between reactive side groups along the copolymer chain(-COOH or -$CONH_2$), to produce side branching and crosslinking of copolymer with a stiffer chain during drying leading to a declining water absorbency.

10. Water absorption capacity of the partially hydrolyzed poly (acrylamide-co-vinyl-2-pyrrolidone) in 0.1, 0.5, 1.0 and 2.0 % w/v of sodium chloride solutions was maximum at 310, 205, 130 and 96 g/g, respectively. An increase in cationic salt content in water reduces the water absorption due to a decrease in the osmotic pressure because the difference in the osmotic pressure between inside and outside the gel decreases.

11. Water absorption capacity of the partially hydrolyzed poly (acrylamide-co-1-vinyl-2-pyrrolidone) in 0.1, 0.5, 1.0 and 2.0 w/v of magnesium chloride solutions was maximum at 153, 96, 63 and 43 g/g, respectively. At the same concentration, magnesium chloride solutions have a

lower absorption value than those of sodium chloride solutions. This suggests that the osmotic pressure equilibrium was reached earlier in the presence of the divalent ion.

As only the total dose can be controlled with the radiation source, it is found that at the fixed dose rate of $1.19 \times 10^4$ Gy/h, total dose 1.4 kGy and concentration of acrylamide 2.5M, the percent conversion of acrylamide monomer is highest. At a fixed dose rate of 0.1990 kGy/min, total dose 10 kGy, 5 g of 71% partially hydrolyzed polyacrylamide and 10 $cm^3$ of 1-vinyl-2-pyrrolidone the water absorption capacity of the copolymer was 950 times their original dried weight. The synergistic effect in increasing the water absorption capacity was further amplified by drying the copolymer with a freeze dryer. The water absorption capacity of the freeze dried method was increased to 1,100 times their original dried weight.

Suggestion and Future Work

There are many techniques to synthesize partially hydrolyzed poly (acrylamide-co-1-vinyl-2-pyrrolidone). The current synthesis technique is the simultaneous irradiation. To develop partially hydrolyzed poly(acrylamide-co-1-vinyl-2-pyrrolidone) by gamma irradiation, which can be used for enhanced oil recovery(EOR) processes through its water absorption, futher work should be carried out as follows:

1. Due to the configurational limitation of the irradiation source of gamma rays, only three dose rates can be adjusted at any one total dose. Intensive research work elsewhere indicated that an optimum dose rate could enhance significantly a copolymerization reaction. Modification of the gamma source to make possible a certain variation in dose rate should be pursued to enable more copolymerization reactions to result in more varieties of EOR superabsorbents.

2. Since the inavailability of solid state NMR, detailed chemical shifts of the three carbonyls of the final products cannot be obtained to confirm the existence of the PVP. Future work should be pursued in this area when the instrument is available.

3. Determination of the average molecular weights of poly-acrylamide, partially hydrolyzed polyacrylamide and poly(acrylamide-co-1-vinyl-2-pyrrolidone) by gel permeation chromatography, should also be carried when a suitable solvent, a pyrolysis or hydrolysis technique of the copolymer/terpolymer are made possible to prepare solution for GFC.

4. Determination of the ratios of acrylamide and acrylate contents in the copolymers should be carried out to correlate with the extent of water absorption.