CHAPTER 2

EXPERIMENTAL TECHNIQUES

2.1 PURIFICATION OF SOLVENTS AND REAGENTS

2.1.1 Solvents

Dimethylacetamide (DMAc), dimethylsulphoxide (DMSO), ethyl acetate, tetrahydrofuran (THF), benzene, chlorobenzene, toluene, chloroform, dichloromethane, 1,4-dioxane, methanol, and ethanol were purified according to standard methods (Vogel’s Text Book 1989) and distilled before use. All other solvents used were purified by distillation.

2.1.2 Triethylamine

Triethylamine (AR, SRL) was distilled and the fraction boiling at 88-89°C was collected and used.

2.1.3 Ethyl methyl ketone (EMK)

To the EMK (LR grade) was added potassium carbonate and kept overnight. Then the EMK solvent was distilled and the fraction boiling at 80°C was collected and used as solvent.
2.1.4 Benzoyl chloride

Benzoyl chloride (BDH) was used as received for the preparation of methacryloyl chloride.

2.1.5 N, N’-Dimethyl formamide (DMF)

DMF (LR grade) was distilled and the fraction boiling at 153ºC was collected and used as a solvent.

2.1.6 Methacrylic acid

Methacrylic acid (CDH) was used as received without removing the inhibitor for the preparation of methacryloyl chloride.

2.1.7 Methyl methacrylate (MMA)

Methyl methacrylate (LR) was freed from inhibitor such as hydroquinone by washing with 5%NaOH solution and distilled water, dried over anhydrous sodium sulphate and distilled under reduced pressure and used for the synthesis of copolymer.

2.1.8 Glycidyl methacrylate (GMA)

Glycidyl methacrylate (Merck) was purified adopting the procedure mentioned for MMA and used for the synthesis of copolymers.
2.1.9 **Benzoyl peroxide (BPO)**

Benzoyl peroxide (LR grade) was recrystallised from chloroform and methanol (1:1 mixture) and used as an initiator.

2.1.10 **Reagents**

p-Amino acetophenone (SRL), p-chlorobenzaldehyde (SRL), 3,4-dimethoxybenzaldehyde (Merck), m and p-nitrobenzaldehyde (Merck), p-N,N'-dimethylaminobenzaldehyde (SRL) were recrystallised from ethanol and dried over vacuum. Benzaldehyde (CDH), m-chlorobenzaldehyde (Merck), m-methyl benzaldehyde (Merck), p-methylbenzaldehyde (SRL), p-methoxy benzaldehyde (Merck), 3-methoxybenzaldehyde (Merck), m and p-bromo benzaldehyde (Merck) 1-naphthaldehyde (SRL) were distilled before use.

2.2 **PREPARATIONS**

2.2.1 **Methacryloyl chloride**

\[
\begin{align*}
\text{CH}_3 & \quad \text{CH}_2=\text{C} \quad + \quad \text{C}_6\text{H}_5\text{COCl} & \quad \text{Distillation} & \quad \text{CH}_3 \\
\text{COOH} & \quad \text{COOH} & \quad 95-97^\circ\text{C} & \quad \text{COOH} \\
\end{align*}
\]
Methacryloyl chloride was prepared according to the method of Stampel et al (1950). A mixture of methacrylic acid (43 g; 0.5 mol), benzoyl chloride (140.5 g; 1 mol) and hydroquinone were taken in 2L round bottom flask and distilled at a fairly rapid rate. The fraction boiling between 95-140°C was collected, redistilled and the fraction boiling between 95-97°C at 740mm was collected. The yield was 19 g (66%).

2.2.2 Aminochalcones

2.2.2.1 4-(3'-Bromocinnamoyl)aniline, (1)

4-(3'-Bromocinnamoyl)aniline (4,3'-BCA) was prepared adopting the procedure of Akelah et al (1992). In a three-necked flask equipped with a mechanical stirrer, thermometer and a dropping funnel, mixed solution of p-aminoacetophenone (10.2 g) in 50 ml of ethanol and sodium hydroxide (3.2 g) in 30 ml of distilled water. It was placed and cooled in an ice-water bath (0-20°C). Then a solution of m-bromobenzaldehyde (9.4 ml) in 20 ml of methanol was added dropwise with constant stirring and the temperature was allowed to exceed 25°C. The stirring was continued for 48 h at room temperature. The crude product was filtered off and washed with cold water. It was then recrystallized from ethyl acetate to get shining yellow crystals. Yield: 14.08 g (67%); mp. 181-182°C. The structure of the compound was confirmed by elemental analysis, FT-IR, 1H-NMR and 13C-NMR spectral techniques.
Elemental analysis (%: C: 59.89 (Found): 59.94 (Calcd.), H: 4.29 (Found): 4.32 Calcd), N: 4.58 (Found): 4.62 (Calcd).

IR (KBr, cm$^{-1}$): 3417 & 3330 (N-H stretching); 3052 (=C-H stretching); 2930 & 2875 (C-H Stretching); 1652 (>C=O); 1608 (olefinic and aromatic >C=C<); 1557 (N-H bending); 1580 & 1442 (aromatic C=C stretching); 1341 & 1304 (C-N stretching); 969 (Ar-Br); 830 & 779 (=C-H out of plane bending); 662 (N-H wagging); 432 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.87-7.24 (m, 9H, Ar-H & =CH-Ar), 6.55-6.51 (d, 1H, CO=CH-), 6.09 (s, 2H, NH$_2$).

$^{13}$C-NMR (DMSO, ppm): 186.60 (keto, >C=O), 154.89-113.64 (Ar-C & -CH=CH-), 142.35 (=CH-Ar), 123.23 (>CO-CH=).

2.2.2.2 4-(4′-Bromocinnamoyl)aniline, (2)

4-(4′-Bromocinnamoyl)aniline (4,4′-BCA) was prepared by the reaction of p-aminoacetophenone (12.2 g) with p-bromobenzaldehyde (11.2ml) in an ethanol-water mixture in the presence of sodium hydroxide (3.6 g) by adopting the procedure as described in 4,3′-BCA system. The product was recrystallized in ethyl acetate to get yellow crystals. Yield: 13.8 g (59%), mp. 161-162°C.

\[ \text{NH}_2 \quad \text{O-CH}_3 \quad + \quad \text{Br} \quad \text{O-H} \quad \xrightarrow{\text{Ethanol / NaOH}} \quad 0 - 20^\circ\text{C} \quad \text{H} \quad \text{O} \quad \text{Br} \quad \text{H} \quad \text{H} \quad \text{NH}_2 \quad + \quad \text{H}_2\text{O} \]
Elemental analysis (%): C: 59.85 (Found) : 59.94 (Calcd), H: 4.27 (Found) : 4.32 (Calcd), N: 4.53 (Found) : 4.62 (Calcd).

FT-IR (KBr, cm⁻¹): 3461 & 3341 (N-H stretching); 3050 (=C-H stretching); 2958 & 2869 (C-H stretching); 1647 (>C=O); 1606 (olefinic and aromatic >C=C<); 1576 (N-H bending); 1548 & 1487 (aromatic C=C stretching); 1344 (C-N stretching); 976 (Ar-Br); 814 & 786 (=C-H out of plane bending); 670 (N-H wagging); 420 (C=C out of plane bending).

¹H-NMR (DMSO, ppm): 7.84-7.45 (m, 9H, Ar-H & =CH-Ar), 6.54-6.50 (d, 1H, -CO-CH=), 6.08 (s, 2H, NH₂).

¹³C-NMR (DMSO, ppm): 186.64 (keto, >C=O), 154.84-113.65 (aromatic carbons &–CH=CH–), 140.84 (=CH-Ar), 124.26 (>CO-CH=).

2.2.2.3 4-(3³-Chlorocinnamoyl)aniline, (3)

4-(3³-Chlorocinnamoyl)aniline (4,3³-CCA) was synthesized by reacting 4-aminoacetophenone (10.8 g) and 3-chlorobenzaldehyde (9.2 ml) in ethanol in the presence of sodium hydroxide (3.2 g) as a base by a similar procedure employed in the preparation of 4,3³-BCA system. The crude product obtained as a yellow powder and recrystallized in ethyl acetate. Yield: 15.72 g (71%); mp. 123-124ºC.

The structure of the compound was confirmed by elemental analysis, FT-IR, ¹H-NMR and ¹³C-NMR spectral techniques.
Elemental analysis (%): C: 69.82 (Found): 69.90 (Calcd), H: 4.64 (Found): 4.69 (Calcd), N: 5.38 (Found): 5.43 (Calcd).

FT-IR (KBr, cm⁻¹): 3419 & 3331 (N-H stretching); 3044 (=C-H stretching); 2933 & 2811 (C-H Stretching); 1628 (>C=O); 1608 (olefinic >C=C<); 1580 (N-H bending); 1514 & 1442 (aromatic C=C stretching); 1341 (C-N stretching); 1021 (Ar-Cl); 830 & 791 (=C-H out of plane bending); 663 (N-H wagging); 502 & 437 (C=C out of plane bending);

¹H-NMR (DMSO, ppm): 7.92-7.34 (m, 9H, Ar-H & =CH-Ar), 6.55-6.51 (d,1H, -CO-CH=), 6.09 (s, 2H, NH₂).

¹³C-NMR (DMSO, ppm): 186.61 (keto, >C=O), 154.89-113.64 (aromatic carbons & –CH=CH–), 140.54 (=CH-Ar), 125.01 (>CO-CH=).

2.2.2.4 4-(4'-Chlorocinnamoyl)aniline, (4)

4-(4'-Chlorocinnamoyl)aniline (4,4'-CCA) has been synthesized by reacting 4-chlorobenzaldehyde (9.8g) with p-aminoacetophenone (9.4 g) in the presence of sodium hydroxide (2.8 g) as a base in an ethanol-water mixer by adopting a similar procedure as described in 4,3'- BCA system. The product was recrystallized in ethyl acetate to get a yellow powder. Yield: 14.3 g (78%); mp. 149-150°C.
The elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectral data of 4,4'-CCA are consistent with the assigned structure.

\[
\text{Ar}^+\text{CH}_3\quad +\quad \text{Cl}\text{Ar}^+\text{CHO}\xrightarrow{\text{Ethanol / NaOH, } 0\text{-}20^\circ C}\quad \text{Cl}\text{Ar}^+\text{CO}-\text{CHAr}^+\text{NH}_2 + \text{H}_2\text{O}
\]

Elemental analysis: C: 69.85 (Found): 69.90 (Calcd), H: 4.60 (Found): 4.69 (Calcd), N: 5.40 (Found): 5.43 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3460 & 3342 (N-H stretching); 3050 (=C-H stretching); 2927 & 2860 (C-H stretching); 1647 (>C=O); 1605 (olefinic >C=C<); 1547 (N-H bending); 1574 & 1490 (aromatic C=C stretching); 1346 (C-N stretching); 1015 (Ar-Cl); 816 & 786 (=C-H out of plane bending); 670 (N-H wagging); 467 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.90-7.28 (m, 9H, Ar-H & =CH-Ar), 6.54-6.51 (d, 1H, -CO-CH=), 6.08 (s, 2H, NH$_2$).

$^{13}$C-NMR (DMSO, ppm):186.62 (keto, >C=O), 154.83 – 113.63 (aromatic carbons & -CH=CH-), 140.76 (=CH-Ar), 124.19 (>CO-CH=).

2.2.2.5 4-(4'-Cinnamoyl)aniline, (5)

4-(4'-Cinnamoyl)aniline (4,4'-CA) was synthesized by the reaction between p-aminoacetophenone (10.5 g) and benzaldehyde (7.86 ml) in the presence of sodium hydroxide (3.2 g) in ethanol-water mixer using the method
similar to that adopted for the preparation of 4,3'‐BCA. The product on recrystallization from ethyl acetate to get pure compound. Yield: 12.34 g (67%); mp. 94-95 °C.

The elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectral data of 4,4'-CA was found to be consistent with the assigned structure.

Elemental analysis (%): C: 80.61 (Found): 80.69 (Calcd), H: 5.82 (Found): 5.87(Calcd), N: 6.23 (Found): 6.27 (Calcd).

IR (KBr, cm$^{-1}$) : 3474 & 3340 (N-H stretching); 3058 & 3026 (=C-H stretching); 2926 & 2844 (C-H Stretching); 1630 (>C=O); 1590 (olefinic and aromatic >C=C<); 1559 (N-H bending); 1494 & 1446 (aromatic C=C stretching); 1304 (C-N stretching); 827 & 766 (=C-H out of plane bending); 673 (N-H wagging); 502 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.84-7.23 (m, 9H, Ar-H), 6.54-6.45 (2d, 2H, -CH=CH-), 6.05 (s, 2H, NH$_2$).

$^{13}$C-NMR (DMSO, ppm): 186.84 (keto, >C=O), 154.76-113.65 (aromatic carbons and –CH=CH–), 142.26(=CH-Ar), 123.34 (>CO-CH=).
2.2.2.6 4-(3',4'-Dimethoxycinnamoyl)aniline, (6)

4-(3',4'-Dimethoxycinnamoyl)aniline (4,3',4'-DMeOCA) was obtained by reacting p-aminoacetophenone (15 g) and 3,4-dimethoxy benzaldehyde (18.44 g) in the presence of sodium hydroxide (4.4 g) in ethanol-water mixer following the procedure for the preparation of 4,3'-BCA. The solid product was recrystallized in ethyl acetate to get yellow crystals. Yield: 20.82 g (62%); mp. 126-127ºC. The structure of the compound 4,3',4'-DMeOCA was confirmed by elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectra.

Elemental analysis (%): C: 74.80 (Found): 74.88(Calcd), H: 6.01 (Found): 6.05(Calcd), N: 4.88 (Found): 4.94 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3449 & 3353 (N-H stretching); 3006 (=C-H stretching); 2922 & 2848 (C-H Stretching); 1643 (>C=O); 1603 (olefinic >C=C<); 1549 (N-H bending); 1603, 1513 & 1445 (aromatic C=C stretching); 1136 (C-N stretching); 1217 &1024 (C-O-C asymmetric and symmetric
stretching); 829 & 793 (=C-H out of plane bending); 685 (N-H wagging); 497 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.68-6.53 (m, 8H, Ar-H & =CH-Ar), 7.85-7.83 (-CO-CH=), 6.00 (s, 2H, NH$_2$), 3.75-3.70 (6H, 2x OCH$_3$).

$^{13}$C-NMR (DMSO, ppm): 186.91 (keto, >C=O), 154.56-113.64 (aromatic carbons & -CH=CH-), 142.76 (=CH-Ar), 124.65 (>CO-CH=), 56.62 & 56.44 (OCH$_3$).

### 2.2.2.7 4-(4'-N,N-Dimethylaminocinnamoyl)aniline, (7)

4-(4'-N,N-Dimethylaminocinnamoyl)aniline (4,4'-DMACA) was prepared by reacting p-aminoacetophenone (8.2 g) and p-N,N-dimethylaminobenzaldehyde (9.05 g) in an ethanol-water mixer and sodium hydroxide as a base by adopting a similar procedure as described in 4,3'-BCA. The product obtained as a orange powder and recrystallized in ethyl acetate. Yield: 12.61 g (74%) mp. 157-158ºC. The structure of 4,4'-DMACA was confirmed by elemental analysis, IR and $^1$H-NMR spectral techniques. The structure of 4,4'-DMACA was confirmed by elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectra.

![Reaction diagram]
Elemental analysis (%): C: 76.60 (Found): 76.66 (Calcd), H: 6.73 (Found): 6.80 (Calcd), N: 10.45 (Found): 10.52 (Calcd).

FT-IR (KBr, cm\(^{-1}\)): 3475 & 3329 (N-H stretching); 3024 (=C-H stretching); 2921 & 2810 (C-H stretching); 1622 (>C=O); 1598 (olefinic and aromatic >C=C<); 1548 (N-H bending); 1527 & 1438 (aromatic, C=C stretching); 1346 (C-N stretching); 808 (=C-H out of plane bending); 682 (N-H wagging); 478 (C=C out of plane bending).

\(^1\)HNMR (DMSO, ppm): 7.88-7.54 (m, 8H, Ar-H), 6.69-6.57 (2d, 2H,-CH), 6.00 (s, 2H, NH\(_2\)), 2.97-2.94 (6H, 2xCH\(_3\)).

\(^1^3\)C-NMR (DMSO, ppm): 186.84 (keto, >C=O), 154.22-113.62 (aromatic carbons &–CH=CH–), 143.36 (=CH-Ar), 123.46 (>CO-CH=), 43.10 (CH\(_3\)).

2.2.2.8 4-(3′-Methoxycinnamoyl)aniline, (8)

4-(3′-Methoxycinnamoyl)aniline (4,3′-MeOCA) was obtained by reacting 3-methoxy benzaldehyde (13.35 ml) and p-aminoacetophenone (14.8 g) in the presence of sodium hydroxide (4.4 g) following the procedure for the preparation of 4,3′-BCA. The product was recrystallized from ethyl acetate to get yellow powder. Yield: 18.4 g (68%); mp, 112-113°C. The elemental analysis, IR, \(^1\)H-NMR and \(^1^3\)C-NMR spectral data of 4,3′-MeOCA was found to be consistent with the assigned structure.
Elemental analysis (%): C: 75.80 (Found): 75.87 (Calcd), H: 5.93 (Found): 5.97 (Calcd), N: 5.48 (Found): 5.53 (Calcd).

FT-IR (KBr, cm⁻¹): 3439 & 3342 (N-H stretching); 3023 (=C-H stretching); 2924 & 2854 (C-H stretching); 1645 (>C=O); 1586 (olefinic and aromatic >C=C<); 1586 (N-H bending); 1442 & 1493 (aromatic C=C stretching); 1340 (C-N stretching); 1256 & 1033 (C-O-C asymmetric and symmetric stretching); 823 & 783 (=C-H out of plane bending); 671 (N-H wagging); 451 (C=C out of plane bending).

¹H-NMR (DMSO, ppm): 7.87-7.26 (m, 9H, Ar-H & =CH-Ar), 6.56-6.54 (d, 1H, -CO-CH=), 6.07 (s, 2H, NH₂), 3.71-3.59 (3H, OCH₃).

¹³C-NMR (DMSO, ppm): 186.89 (keto, >C=O), 154.79-113.65 (aromatic carbons & -CH=CH-), 142.32 (=CH-Ar), 123.63 (>CO-CH=), 56.13 (-OCH₃).

2.2.2.9 4-(4′-Methoxycinnamoyl)aniline, (9)

4-(4′-Methoxycinnamoyl)aniline (4,4′-MeOCA) has been synthesized by reacting p-aminoacetophenone (9.8 g) with p-methoxy benzaldehyde (8.8 ml) in the presence of sodium hydroxide (2.5 g) in a water-ethanol mixer by a method similar to 4,3′-BCA system. The crude compound was recrystallized in methanol to get a yellow powder. Yield: 11.89 g (64%); mp. 81-82°C. The structure of 4,3′-MeOCA was confirmed by elemental analysis, IR, ¹H-NMR and ¹³C-NMR spectral techniques.
Elemental analysis (%): C: 75.84 (Found); 75.87 (Calcd), H: 5.90 (Found); 5.97 (Calcd), N: 5.46 (Found); 5.53 (Calcd).

FT-IR (KBr, cm⁻¹): 3441 & 3347 (N-H stretching); 3012 (=C-H stretching); 2930 & 2837 (C-H stretching); 1651 (>C=O); 1600 (olefinic and aromatic >C=C<); 1510 (N-H bending); 1600 & 1440 (aromatic C=C stretching); 1340 (C-N stretching); 1230 & 1029 (C-O-C asymmetric and symmetric stretching); 822 & 749 (=C-H out of plane bending); 676 (N-H wagging); 418 (C=C out of plane bending).

¹H-NMR (DMSO, ppm): 8.11-7.26 (m, 8H, Ar-H), 6.95 – 6.53 (2d, 2H, -CH=CH-), 6.02 (s, 2H, NH₂), 3.72-3.69 (OCH₃).

¹³C-NMR (DMSO, ppm): 186.88 (keto, >C=O), 154.55 – 114.88 (aromatic carbons & –CH=CH-), 142.26 (=CH-Ar), 120.42 (>CO-CH=), 56.17 (OCH₃).

2.2.2.10 4-(3′-Methylcinnamoyl)aniline, (10)

4-(3′-Methylcinnamoyl)aniline (4,3′-MCA) was prepared by reacting p-aminoacetophenone (14 g) with 3-methyl benzaldehyde (12.2 ml) in the presence of sodium hydroxide (4 g) in water-ethanol mixer by a method similar to the procedure adopted in 4,3′-BCA system. The product obtained as
yellow powder recrystallized in ethyl acetate. Yield: 17.06 g (66%); mp. 96-97\(^\circ\)C. The elemental analysis, IR, \(^1\)H-NMR and \(^{13}\)C-NMR spectral data of 4,3'-MCA was found to be consistent with the assigned structure.

Elemental analysis (%): C: 80.94 (Found): 80.98 (Calcd), H: 6.31 (Found): 6.37 (Calcd), N: 5.87 (Found): 5.90 (Calcd).

FT-IR (KBr, cm\(^{-1}\)): 3479 & 3334 (N-H stretching); 3060 (=C-H stretching); 2922 & 1884 (C-H stretching); 1621 (>C=O); 1600 (olefinic and aromatic >C=C<); 1556 (N-H bending); 1600 & 1443 (aromatic C=C stretching); 1340 (C-N stretching); 835 & 790 (=C-H out of plane bending); 674 (N-H wagging); 499 (C=C out of plane bending).

\(^1\)H-NMR (DMSO, ppm): 7.94-7.21 (m, 9H, Ar-H & =CH-Ar), 6.64-6.62 (d, 1H, CO-CH=), 6.14 (s, 2H, NH\(_2\)), 2.39-2.35 (-CH\(_3\)).

\(^{13}\)C-NMR (DMSO, ppm): 186.83 (keto, >C=O), 154.73 – 113.65 (aromatic carbons & –CH=CH_), 142.26 (=CH-Ar), 123.34 (>CO-CH_), 21.75 (-CH\(_3\)).

2.2.2.11 4-(4'-Methylcinnamoyl)aniline, (11)

4-(4'-Methylcinnamoyl)aniline (4,4'-MCA) was synthesized by reacting p-aminoacetophenone (10.5 g) with p-methylbenzaldehyde (9.2 ml) in an ethanol-water mixer in the presence of sodium hydroxide (3 g) by adopting the procedure as described in 4,3'-BCA system. The product was recrystallized
from ethyl acetate to get yellow powder. Yield: 14.82 g (78%); mp. 146-147°C. The structure of 4,4'-MCA was confirmed by elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectral techniques.

Elemental analysis (%): C: 80.92 (Found): 80.98 (Calcd), H: 6.34 (Found): 6.37 (Calcd), N: 5.85 (Found): 5.90 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3461 & 3341 (N-H stretching); 3026 (=C-H stretching); 2917 & 2860 (C-H stretching); 1628 (>C=O); 1598 (olefinic and aromatic >C=C<); 1515 (N-H bending); 1598 & 1442 (aromatic C=C stretching); 1341 (C-N stretching); 811 & 741 (=C-H out of plane bending); 676 (N-H wagging); 502 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.81-7.00 (m, 9H, Ar-H & =CH-Ar), 6.52-6.50 (d, 1H, -CO-CH=), 6.01 (s, 2H, N-H$_2$), 2.39-2.13 (b, -CH$_3$).

$^{13}$C-NMR (DMSO, ppm): 186.89 (keto, >C=O), 154.31-113.60 (aromatic carbons & -CH=CH-), 142.65 (=CH-Ar), 123.13 (>CO-CH=), 21.92 (CH$_3$).
2.2.2.12 4-(3′-Nitrocinnamoyl)aniline, (12)

4-(3′-Nitrocinnamoyl)aniline (4,3′-NCA) was obtained by reacting p-aminoacetophenone (12.5 g) with 3-nitrobenzaldehyde (13.8 g) in the presence of sodium hydroxide (3.7 g) in an ethanol-water mixer according to the procedure employed for the preparation of 4,3′-BCA. The solid product obtained as recrystallised in ethyl acetate to get a pure yellow powder. Yield: 17.64 g (71%); mp. 209-210ºC. The elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectral data of 4,3′-NCA was found to be consistent with the assigned structure.

Elemental analysis (%): C: 67.10 (Found): 67.16 (Calcd), H: 4.48 (Found): 4.51 (Calcd), N: 10.40 (Found): 10.44 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3427 & 3338 (N-H stretching); 3065 (=C-H stretching); 2924 & 2856 (C-H stretching); 1633 (>
H=O); 1609 (olefinic >C=C<); 1556 (N-H bending); 1580, 1531 & 1444 (aromatic C=C stretching); 1345 (C-N stretching); 1233 (NO$_2$); 830 & 791 (=C-H out of plane bending); 656 (N-H wagging); 513 (C=C out of plane bending).

$^1$H-NMR (DMSO, ppm): 7.96-7.63 (m, 9H, Ar-H & =CH-Ar), 6.63 –6.60 (d, 1H, -CO-CH=), 6.19 (s, 2H, N-H2).
$^{13}$C-NMR (DMSO, ppm): 186.42 (Keto, >C=O), 155.01-113.62 (aromatic carbons & –CH=CH–), 139.75 (=CH-Ar), 125.95 (>CO-CH=).

### 2.2.2.13 4-(4′-Nitrocinnamoyl)aniline, (13)

4-(4′-Nitrocinnamoyl)aniline (4,4′-NCA) has been synthesized following the procedure for the preparation of 4,3′-BCA by reacting p-aminoacetophenone (10.4 g) and p-nitrobenzaldehyde (11.6 g) in the presence of sodium hydroxide (3.2 g) in water-ethanol mixer. The crude compound was recrystallized from ethyl acetate. Yield: 16.03 g (76%); mp. 208-209ºC.

The structure of 4,4′-NCA was confirmed by elemental analysis, IR, $^1$H-NMR and $^{13}$C-NMR spectral techniques.

![Reaction Scheme]

Elemental analysis (%): C: 67.13 (Found): 67.16 (Calcd), H: 4.45 (Found): 4.51 (Calcd), N: 10.38 (Found): 10.44 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3485 & 3388 (N-H stretching); 3107 (=C-H stretching); 2925 & 2778 (C-H Stretching); 1637 (>C=O); 1595 (olefinic and aromatic >C=C<); 1508 (N-H bending); 1595 & 1441 (aromatic C=C stretching); 1341 (C-N stretching); 1230 (NO$_2$); 828 & 758 (=C-H out of plane bending); 669 (N-H wagging); 502 (C=C out of plane bending).
$^1$H-NMR (DMSO, ppm): 8.18-7.62 (m, 9H, Ar-H & =CH-Ar), 6.62-6.60 (d, 1H, -CO-CH=), 6.22 (s, 2H, NH$_2$).

$^{13}$C-NMR (DMSO, ppm: 186.26 (keto, >C=O), 155.11-113.65 (aromatic carbons & -CH=CH-), 142.66 (=CH-Ar), 125.87 (>CO-CH=).

2.2.2.14 4-(2',3'-Benzocinnamoyl)aniline, (14)

4-(2',3'-Benzocinnamoyl)aniline (4,2'-BCA) was prepared by reacting p-aminoacetophenone (8.8 g) with 1-naphthaldehyde (8.9 ml) in the presence of sodium hydroxide (2.4 g) in water-methanol mixer adopting the procedure as described in 4,3'-BCA. The product obtained as yellow powder and recrystallized in ethyl acetate. Yield: 13.07 g (72%); mp. 194-195ºC.

The elemental analysis, IR and $^1$H-NMR and $^{13}$C-NMR spectral data of 4,2',3'-BCA was found to be consistent with the assigned structure.

Elemental analysis (%): C: 83.46 (Found): 83.50 (Calcd), H: 5.45 (Found): 5.53 (Calcd), N: 5.08 (Found): 5.12 (Calcd).

FT-IR (KBr, cm$^{-1}$): 3408 & 3327 (N-H stretching); 3044 (C-H stretching); 2929 & 2814 (C-H Stretching); 1637 (>C=O); 1607 (olefinic
>C=C<); 1560 (N-H bending); 1514 & 1443 (aromatic C=C stretching); 1350 (C-N stretching); 830 & 788 (=C-H out of plane bending); 685 (N-H wagging); 496 & 439 (C=C out of plane bending).

\[ ^1H \text{-NMR (DMSO, ppm)} : 8.48-8.40 \text{ (d, 1H), 8.23-8.13 \text{ (2d, 2H), 8.00-7.89 (m, 5H), 7.60-7.51 (m, 3H, aromatic), 6.6-6.66 (d, 1H, =CH-Ar), 6.21-6.19 (d, 1H, =CH=CO), 3.43 (s, 2H, NH}_2). \]

\[ ^{13}C \text{-NMR (DMSO, ppm: 186.73 (Keto, >C=O), 154.90-113.75 (aromatic carbons & -CH=CH-)} 138.30 (=CH-Ar), 126.07 (>CO-CH=). \]

2.2.3 Monomers

2.2.3.1 4-(3′-Bromocinnamoyl)phenyl methacrylamide, (15)

\[ \text{H}_2\text{N} + \text{H} \rightarrow \text{H}_2\text{N} \]

\[ \text{H} \rightarrow \text{H} \text{Cl} \rightarrow \text{H} \text{CH}_3 \]

4-(3′-Bromocinnamoyl)phenyl methacrylamide (3-BCPM) was synthesized adopting the procedure of Rami Reddy et al (1992). 4-(3′-Bromocinnamoyl) aniline (8.8 g, .03 mol), triethylamine (4.06 ml, .03 mol) and ethyl methyl ketone (EMK) (250 ml) were taken in a three necked round bottom flask. A mechanical stirrer was attached to the center neck, a dropping funnel with pressure equalising arrangement was attached to the second neck
and a guard tube was attached to the third neck. Methacryloyl chloride (2.8 ml, 0.03 mol) in 25 ml of EMK was taken in the dropping funnel. The RB flask was placed in an ice bath and the contents were stirrer well. Methacryloyl chloride was added dropwise with constant stirring. The reaction mixer was stirred in cold condition for 2 hrs and then at room temperature for 1 h. The solid triethyl ammonium chloride was removed by filtration and the solvent in the filtrate was removed using rotary evaporator to get crude 4-(3′-bromo cinnamoyl)phenyl methacrylamide. The residue obtained was extracted with chloroform and the solution was washed successively with distilled water to remove the unreacted materials. Finally the chloroform layer dried over anhydrous sodium sulphate and then it was evaporated using rotary evaporator. The solid product was recrystallized in methanol-ethyl acetate mixer to get yellow crystals. Yield: 8.79 g (76 %); mp. 139-140ºC.

2.2.3.2 4-(4′-Bromocinnamoyl)phenyl methacrylamide, 16)

4-(4′-Bromocinnamoyl)phenyl methacrylamide (4-BCPM) was synthesized by reacting 4-(4′-bromocinnamoyl) aniline (8 g, 0.03 mol) and methacryloyl chloride (2.7 ml, 0.03 mol) in the presence of triethylamine (3.7 ml, 0.03 mol) as acid scavenger in ethyl methyl ketone by a similar procedure as described in the preparation of 4-(3′-bromocinnamoyl)phenyl methacrylamide. The product was recrystallized from ethyl acetate to get a yellow powder. Yield: 8.49 g (79 %); mp.151-152ºC.
2.2.3.3 4-(3'-Chlorocinnamoyl)phenyl methacrylamide, (17)

\[
\text{H}_2\text{N} \quad \text{H} \quad \text{H} \quad \text{Cl} \quad \text{H} \quad \text{O} \\
\text{H} \quad \text{H} \quad \text{C} \quad \text{H} \quad \text{3} \quad \text{Cl} \\
\text{H} \quad \text{C} \quad \text{H} \quad \text{3} \quad \text{O} \\
\text{H} \quad \text{H} \quad \text{Cl} \quad \text{Et}_3\text{N} \\
\text{Et}_3\text{NHCl}
\]

4-(3'-Chlorocinnamoyl)phenyl methacrylamide (3-CCPM) was obtained by reacting 4-(3'-chlorocinnamoyl)aniline (16 g, 0.06 mol) and methacryloyl chloride (6 ml, 0.06 mol) in the presence of triethylamine (8.7 ml, 0.06 mol) in EMK by adopting a similar procedure as described in the preparation of 3-BCPM. The crude product was recrystallized from ethyl acetate to get yellow powder. Yield: 16.89 g (77 %); mp.138-139 °C.

2.2.3.4 4-(4'-Chlorocinnamoyl)phenyl methacrylamide, (18)

4-(4'-Chlorocinnamoyl)phenyl methacrylamide (4-CCPM) was synthesized by reacting 4-(4'-chloro cinnamoyl)aniline (8 g, 0.03 mol) with
methacryloyl chloride (3 ml, 0.03 mol) in the presence of triethylamine (4.3 ml, 0.03 mol) in ethyl methyl ketone under similar conditions as explained in the synthesis of 3-BCPM. The crude product was recrystallized in EMK-ethyl acetate mixer to get a yellow powder. Yield: 8.01 g (73%); mp. 160-161ºC.

\[ \text{H}_2\text{N} \begin{array}{c} \text{H} \\ \text{H} \\ \text{C} \end{array} \begin{array}{c} \text{H} \\ \text{H} \\ \text{Cl} \end{array} \text{O} + \text{H} \begin{array}{c} \text{CH}_3 \\ \text{Cl} \\ \text{C} \end{array} \text{H} \text{Cl} \xrightarrow{0-20\,^\circ\text{C}} \text{Et}_3\text{N} + \begin{array}{c} \text{H} \\ \text{H} \\ \text{Cl} \end{array} \begin{array}{c} \text{H} \\ \text{H} \\ \text{Cl} \end{array} \text{O} \text{N} \text{H} \text{O} \text{H} \text{Cl} + \text{Et}_3\text{NCl} \\

2.2.3.5 4-(4’-Cinnamoyl)phenyl methacrylamide, (19)

4-(4’-Cinnamoyl)phenyl methacrylamide (CPM) was obtained by reacting 4-cinnamoyl aniline (12.1 g, 0.05 mol) with methacryloyl chloride (5.25 ml, 0.05 mol) in EMK in the presence of triethylamine (7.56 ml, 0.05 mol) following the same procedure described in the preparation of 3-BCPM.
The monomer was recrystallized in methanol-ethyl acetate (1:1) mixer to get 14.16 g (81%) of yellow powder mp. 83-84°C.

2.2.3.6 4-(3',4'-Dimethoxycinnamoyl)phenyl methacrylamide, (20)
4-(3',4'-Dimethoxycinnamoyl)phenyl methacrylamide (DMeOCPM) was prepared by reacting 4-(3',4'-Dimethoxycinnamoyl) aniline (16 g, 0.06 mol) and methacryloyl chloride (5.5 ml, 0.06 mol) in EMK in the presence of triethylamine (7.9 ml, 0.06 mol) by using a similar procedure as described in the preparation of 3-BCPM. The product was recrystallized in ethyl acetate to get yellow shining crystals. Yield: 17.81 g (83 %); mp. 60-61°C.

2.2.3.7 4-(4'-N,N-dimethylethylaminocinnamoyl)phenyl methacrylamide, (21)

4-(4'-N,N-dimethylethylaminocinnamoyl)phenyl methacrylamide (DMCPM) was synthesized by reacting 4-(4'-N,N-dimethylamino cinnamoyl)aniline (8 g, 0.03mol) with methacryloyl chloride (2.9 ml, 0.03mol) in chloroform in the presence of triethylamine (4.2 ml, 0.03 mol) following the same procedure as described in the preparation of 3-BCPM. The product was washed thoroughly with distilled water and dried over anhydrous sodium.
sulphate. Then the chloroform layer was evaporated using rotary evaporator to get a pure monomer. Yield: 7.53 g (69 %); mp. 154-155°C.

2.2.3.8 4-(3′-Methoxycinnamoyl)phenyl methacrylamide, (22)
4-(3′-Methoxycinnamoyl)phenyl methacrylamide  (3-MeOCPM)
was obtained by the reaction between 4-(3′-methoxycinnamoyl)aniline (16 g, 0.06 mol) and methacryloyl chloride (6.1 ml, 0.06 mol) in EMK in the presence of triethylamine (8.8 ml, 0.06 mol) by adopting a similar procedure as described in the preparation of 3-BCPM. The product was recrystallized in methanol-ethyl acetate mixer to get a yellow powder. Yield: 15.91 g (72 %); mp. 121-122ºC.

2.2.3.9  4-(4′-Methoxycinnamoyl)phenyl methacrylamide, (23)
4-(4′-Methoxycinnamoyl)phenyl methacrylamide (4-MeOCPM) was prepared by reacting 4-(4′-methoxycinnamoyl) aniline (8 g, 0.03 mol) with methacryloyl chloride (3.06 ml, 0.03 mol) in the presence of triethylamine (4.4 ml, 0.03 mol) under similar conditions adopted in the preparation of 3-BCPM. The product obtained as a semi solid and extracted in chloroform solution. Then it was washed successively with distilled water and the chloroform layer was dried over anhydrous sodium sulphate. Then the solvent was evaporated using rotary evaporator to get a pure monomer. Yield: 8.42 g (75 %). mp. 59-60°C.

2.2.3.10 4-(3′-Methylcinnamoyl)phenyl methacrylamide, (24)

4-(3′-Methylcinnamoyl)phenyl methacrylamide (3-MCPM) was synthesized by reacting 4-(3′-methylcinnamoyl) aniline (16 g, 0.07 mol) with methacryloyl chloride (6.5 ml, 0.07 mol) in the presence of triethylamine (10.4 ml, 0.07 mol) in EMK by a similar procedure as described in the preparation of 3-BCPM. The crude monomer was recrystallized in ethyl acetate to get a yellow powder. Yield: 18.53 g (82 %), mp. 73-74°C.
2.2.3.11 4-(4'-Methylcinnamoyl)phenyl methacrylamide, (25)

4-(4'-Methylcinnamoyl)phenyl methacrylamide (4-MCPM) was obtained by reacting 4-(4'-methylcinnamoyl)aniline (15 g, 0.06 mol) with methacryloyl chloride (6.1 ml, 0.06 mol) in the presence of triethylamine (8.8 ml, 0.06 mol) in EMK using the method followed in the synthesis of 3-BCPM. The crude monomer was recrystallized from ethyl acetate to get a pure compound. Yield: 15.94 (76 %); mp. 142-143°C.
2.2.3.12 4-(3'-Nitrocinnamoyl)phenyl methacrylamide, (26)

4-(3'-Nitrocinnamoyl)phenyl methacrylamide (3-NCPM) was synthesized by reacting 4-(3'-nitrocinnamoyl)aniline (9.5 g, 0.04 mol) with methacryloyl chloride (3.4 ml, 0.04 mol) in the presence of triethylamine (4.9 ml, 0.04 mol) in EMK by adopting a similar procedure as described in the preparation of 3-BCPM. The product was recrystallized in EMK–ethyl acetate to get a yellow powder. Yield: 9.58 g (74 %); mp. 162-163°C.

\[ 
\text{H}_2\text{N} \quad \text{H} \quad \text{H} \quad \text{NO}_2 \quad \text{O} \quad \text{H} 
+ \quad \text{H} \quad \text{CH}_3 \quad \text{H} \quad \text{Cl} \quad \text{O} \quad \text{Cl} 
\overset{\text{EMK, Et}_3\text{N}}{\underset{0 - 20 \ ^\circ\text{C}}{\longrightarrow}} 
\text{H} \quad \text{CH}_3 \quad \text{H} \quad \text{NO}_2 \quad \text{O} \quad \text{H} 
+ \quad \text{Et}_3\text{NCl} 
\]

2.2.3.13 4-(4'-Nitrocinnamoyl)phenyl methacrylamide, (27)

4-(4'-Nitrocinnamoyl)phenyl methacrylamide (4-NCPM) was obtained by reacting 4-(4'-nitrocinnamoyl)aniline (10 g, 0.04 mol) with methacryloyl chloride (3.6 ml, 0.04 mol) in EMK in the presence of...
triethylamine (5.2 ml, 0.04 mol) using the same procedure followed in the preparation of 3-BCPM. The product was recrystallized in EMK-ethyl acetate mixer to get a pure monomer. Yield: 10.49 g (79 %); mp. 187-188ºC.

2.2.3.14 4-(2',3'-Benzocinnamoyl)phenyl methacrylamide, (28)

4-(2’,3’-Benzocinnamoyl)phenyl methacrylamide (BCPM) was prepared by reacting 4-(2’,3’-benzocinnamoyl)aniline (10 g, 0.04 mol) with
methacryloyl chloride (3.6 ml, 0.04 mol) in the presence of triethylamine (5.1 ml, 0.04 mol) in EMK by adopting a similar procedure as described in the preparation of 3-BCPM. The product was recrystallized in ethyl acetate to get a pure monomer. Yield: 10.75 g (79 %); mp. 133-134°C.

The expected structures of monomers (15-28) were confirmed by elemental analysis, IR, $^1$H and $^{13}$C-NMR spectral techniques and these data are presented under Chapter 3, Results and Discussion.

2.3 HOMOPOLYMERISATION

Homopolymerization of the prepared methacrylic monomers was carried out in pyrex glass polymerization tube containing an inlet and outlet for the passage of nitrogen. All the synthesized monomers were polymerized as 2M solutions in EMK using benzoyl peroxide (BPO) (0.5wt% with respect to monomer) as a free radical initiator. Predetermined quantities of the monomer, BPO and the solvent were mixed in a polymerization tube purged with oxygen free N$_2$ gas for 20 min and the tube was then kept in a thermostat maintained at 70°±1°C. After 24 hours, the contents were poured into excess methanol to isolate the polymer. The precipitated polymer was filtered off and further purified by repeated reprecipitation in methanol from a solution of the polymer in methanol in EMK and finally dried under vacuum at 50°C for 24 hours. The following homopolymers were prepared.
<table>
<thead>
<tr>
<th>S.No</th>
<th>Polymers</th>
</tr>
</thead>
<tbody>
<tr>
<td>29</td>
<td>Poly[4-(3'-bromocinnamoyl)phenyl methacrylamide] [Poly(3-BCPM)]</td>
</tr>
<tr>
<td>30</td>
<td>Poly[4-(4'-bromocinnamoyl)phenyl methacrylamide] [Poly(4-BCPM)]</td>
</tr>
<tr>
<td>31</td>
<td>Poly[(3-chlorocinnamoyl)phenyl methacrylamide] [Poly(3-CCPM)]</td>
</tr>
<tr>
<td>32</td>
<td>Poly[4-(4'-chlorocinnamoyl)phenyl methacrylamide] [Poly(4-CCPM)]</td>
</tr>
<tr>
<td>33</td>
<td>Poly[4-(4'-cinnamoyl)phenyl methacrylamide] [Poly(4-CPM)]</td>
</tr>
<tr>
<td>34</td>
<td>Poly[4-(3',4'-Dimethoxycinnamoyl)phenyl methacrylamide] [Poly( DMeOCPM)]</td>
</tr>
<tr>
<td>35</td>
<td>Poly[4-(4'-N,N-Dimethylamino cinnamoyl)phenyl methacrylamide] [Poly(4- DMCPM)]</td>
</tr>
<tr>
<td>36</td>
<td>Poly[4-(3'-methoxycinnamoyl) phenyl methacrylamide] [Poly(3-MeOCPM)]</td>
</tr>
<tr>
<td>37</td>
<td>Poly[4-(4'-methoxycinnamoyl)phenyl methacrylamide] [Poly(4-MeOCPM)]</td>
</tr>
<tr>
<td>38</td>
<td>Poly[4-(3'-methylcinnamoyl) phenyl methacrylamide] [Poly(3-MCPM)]</td>
</tr>
<tr>
<td>39</td>
<td>Poly[4-(4'-methylcinnamoyl)phenyl methacrylamide]</td>
</tr>
</tbody>
</table>
[Poly(4-MCPM)]

40 Poly[4-(3’-nitrocinnamoyl)phenyl methacrylamide]

[Poly(3-NCPM)]

41 Poly[4-(4’-nitrocinnamoyl)phenyl methacrylamide]

[Poly(4-NCPM)]

42 Poly[4-(2’,3’-benzocinnamoyl)phenyl methacrylamide]

[Poly(BCPM)]

2.4 COPOLYMERIZATION

2.4.1 Copolymers of substituted methacrylamides with MMA

Copolymers of substituted methacrylamides with methyl methacrylate (MMA) were synthesized by free radical solution polymerization. The copolymers of these monomers with different feed composition were synthesized in EMK at 70 °C ± 1°C in the presence of BPO as a free radical initiator. In all the cases, the initial total monomers concentration was 1 mol/L and the amount of initiator (BPO) was 0.5wt% based on the monomers. Predetermined quantities of the monomers, BPO and EMK were mixed in a reaction tube and flushed with oxygen free N₂ gas for 20 minutes to maintain inert atmosphere. The sealed tube were then kept in a thermostat at 70°F±1°C. The conversions were restricted to less than 10% in order to satisfy the copolymerization equation. After the required time, the reaction mixture was poured into excess methanol to isolate the polymer. The copolymer was further purified by repeated precipitation by methanol from a solution of the polymer in EMK and finally dried in vaccum at 40°C for constant weight. The
following substituted methacrylamides copolymerized with MMA were synthesized.

<table>
<thead>
<tr>
<th>S.NO</th>
<th>Copolymers</th>
</tr>
</thead>
<tbody>
<tr>
<td>43</td>
<td>Poly[4-(3'-chlorocinnamoyl)phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly(3-CCPM-\textit{co}-MMA)]</td>
</tr>
<tr>
<td>44</td>
<td>Poly[4-(cinnamoyl)phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly(CPM-\textit{co}-MMA)]</td>
</tr>
<tr>
<td>45</td>
<td>Poly[4-(3',4'-dimethoxycinnamoyl)phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly( DMeOCPM-\textit{co}-MMA)]</td>
</tr>
<tr>
<td>46</td>
<td>Poly[4-(4'-methoxycinnamoyl)phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly(4- MeOCPM-\textit{co}-MMA)]</td>
</tr>
<tr>
<td>47</td>
<td>Poly [4-(3'-methylcinnamoyl) phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly(3-MCPM-\textit{co}-MMA)]</td>
</tr>
<tr>
<td>48</td>
<td>Poly [4-(4'-methylcinnamoyl) phenyl methacrylamide-\textit{co}-methyl methacrylate] [Poly(4-MCPM-\textit{co}-MMA)]</td>
</tr>
</tbody>
</table>

### 2.4.2 Copolymers of substituted methacrylamides with GMA

Copolymers of substituted methacrylamides, 3-CCPM, DMeOCPM, 3-MeOCPM, 3-MCPM and 3-NCPM with glycidyl methacrylate (GMA) were synthesized by adopting the same procedure as described in Section 2.4.1. For
each of the copolymer systems six different feed compositions of the monomers were taken. The following copolymers were prepared.

<table>
<thead>
<tr>
<th>S.NO</th>
<th>Copolymers</th>
</tr>
</thead>
<tbody>
<tr>
<td>49</td>
<td>Poly[(3-chlorocinnamoyl)phenyl methacrylamide-\textit{co}-glycidyl methacrylate], [Poly(3-CCPM-\textit{co}-GMA)]</td>
</tr>
<tr>
<td>50</td>
<td>Poly[4-(3',4'-Dimethoxycinnamoyl)phenyl methacrylamide-\textit{co}-glycidyl methacrylate], [Poly(DMeOCPM-\textit{co}-GMA)]</td>
</tr>
<tr>
<td>51</td>
<td>Poly[4-(3'-methoxycinnamoyl)phenyl methacrylamide-\textit{co}-glycidyl methacrylate], Poly(3-MeOCPM-\textit{co}-GMA)]</td>
</tr>
<tr>
<td>52</td>
<td>Poly [4-(3'-methylcinnamoyl) phenyl methacrylamide-\textit{co}-glycidyl methacrylate], [Poly(3-MCPM-\textit{co}-GMA)]</td>
</tr>
<tr>
<td>53</td>
<td>Poly[4-(3'-nitrocinnamoyl)phenyl methacrylamide-\textit{co}-glycidyl methacrylate], [Poly(3-NCPM-\textit{co}-GMA)]</td>
</tr>
</tbody>
</table>

2.5 **ANALYTICAL TECHNIQUES**

2.5.1 **Elemental Analysis**

Micro elemental analysis of the monomers and polymers was performed with Perkin-Elmer 240C- CHN Elemental Analyzer.
2.5.2 Solubility

The solubility of the polymers was tested in several solvents in cold condition. About 0.2 g of the polymer was taken in 10 ml of the solvent and kept overnight in a closed test tube. The solubility was tested after 24 h.

2.5.3 Ultraviolet Spectra

UV spectra of the homopolymers and copolymers and their repeat spectra after irradiation with UV lamp were obtained with Shimadzu UV-1601 UV-visible spectrophotometer.

2.5.4 Infrared Spectra

The FT-IR spectra of the chalcones, methacrylamide monomers, their homopolymers and copolymer samples were recorded in Nicolet Avator 60 FT-IR spectrophotometer using KBr pellets and the liquid monomers in NaCl plates.

2.5.5 $^1$H - NMR Spectra

$^1$H-NMR spectra of the monomers and polymers were run on Hitachi 400 MHZ NMR spectrometer. The spectra were recorded at room temperature as 15-20%(w/v) solutions in CDCl3. Tetramethylsilane (TMS) was used as the internal references.
2.5.6  $^{13}$C - NMR Spectra

$^{13}$C - NMR spectra were recorded on Bruker DRX 125.77 MHz FT-NMR spectrometer. Samples were examined using 10-15% (w/v) solutions in CDCl$_3$ using tetramethylsilane (TMS) as the internal standard.

2.5.7  Gel permeation chromatography

The number ($M_n$) and weight ($M_w$) average molecular weights of the polymers were determined with Waters 501 gel permeation chromatography equipped with three ultra styrigel columns and a differential refractive index (RI-401) detector. The molecular weights were calibrated against polystyrene standards using tetrahydrofuran as mobile phase.

2.5.8  Differential Scanning Calorimetry (DSC)

The glass transition temperature ($T_g$) of the polymers was determined with NETZSCH-Geratebau GmbH DSC 204 thermal analyzer. Samples of 10 mg were used at a temperature rise of 10 °C/min in N$_2$ atmosphere.

2.5.9  Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was carried out on Mettler TA 3000 thermal analyzer. TG traces were recorded on 10 mg samples in air atmosphere at a heating rate of 20 °C.
2.6 PHOTO CROSSLINKING STUDIES

A medium-pressure mercury lamp (Heber Scientific Photoreactor – UV, 6W, 254nm) was used as the UV source for studying the photoreactivity of the synthesized photosensitive polymers. The chloroform solution of the polymers were irradiated (in quartz cell) at a distance of 10cm from the light source for different intervals of time. After each exposure, the UV spectra were recorded at different intervals and the rate of photocrosslinking was followed by a decrease in UV absorption intensity at a suitable wavelength.