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NOTE
NEW ORGANIC POLYMERS. 5. SYNTHESIS OF POLY(2-VINYL-3-SUBSTITUTED-4-QUINAZOLONE)S

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INTRODUCTION

Heterocyclic polymers are well known for their outstanding thermal stability [1]. Sillion et al. [2, 3] reported that polyquinazolones prepared from 6,6′-bi(3,1-benzoxazin-4-one) substituted in the 2-position by a methyl or phenyl group and aromatic diamines are soluble in *m*-cresol and possess good thermal stability. The present paper deals with the synthesis of polyquinazolones from poly[1-(2-carboxyanilinocarbonyl)ethylene]. See Figs. 1 and 2.

EXPERIMENTAL

Poly[1-2-carboxyanilinocarbonyl)ethylene] (**1**) was prepared and characterized according to the literature [4]. Poly[1-(4-oxo-3,1-benzoxazin-2-yl)-ethylene] (**2**) was prepared according to the literature [5] from **1** (5 g) by refluxing in acetic anhydride (200 mL) for 4 h with occasional stirring. The resulting pale yellow solution was cooled while the product precipitated out. The solid was filtered, washed thoroughly with petroleum ether (40-60),

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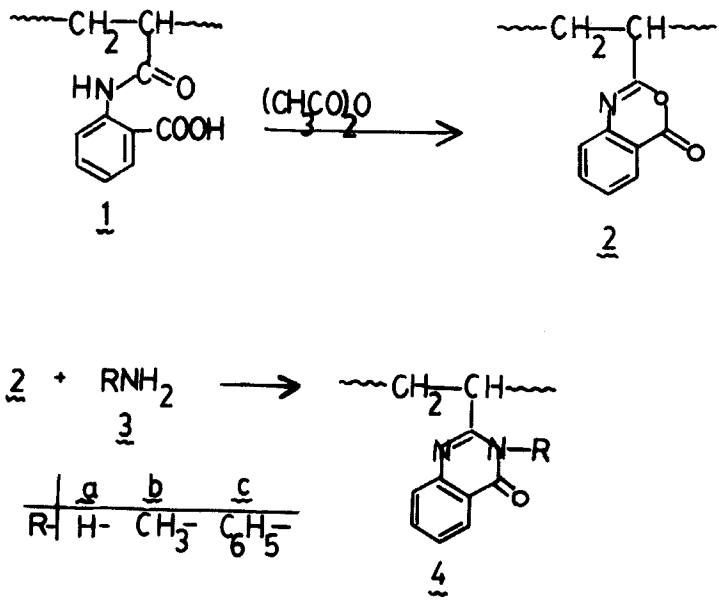

FIGURE 1.

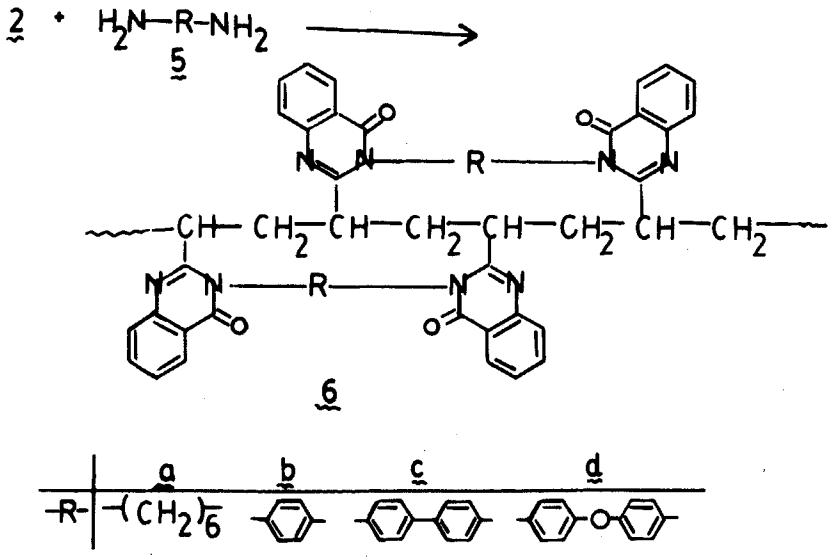

FIGURE 2.

and dried. Yellow crystalline powder; softening point: 170-172°C; yield: 70%. $(C_{10}H_7NO_2)_n$, $(173)_n$. Calculated: N, 8.09%. Found: N, 8.12%.

Polycondensation with Monoamines. Poly[1-(4-oxo-2-quinazolinyl)-ethylene] **(4a)**: **2** (1.2 g) and liquor ammonia (5 mL) were mixed and heated on boiling water bath for 4 h. The residue obtained was filtered, washed with water, and finally with methanol. It was further heated with polyphosphoric acid (Reidel, Germany) (10 g) at 170°C for 8 h, and the viscous solutions was dropped into water (300 mL). The solid mass was filtered, washed with aqueous sodium hydrogen carbonate solution (5%) and then distilled water, and dried at 60°C. It does not melt up to 360°C. Characteristic data are given in Table 1.

Poly[1-(3-methyl-4-oxo-2-quinazolinyl)ethylene] **(4b)** and poly[1-(3-phenyl-4-oxo-2-quinazolinyl)ethylene] **(4c)** were prepared from **2** with methylamine and aniline, respectively, by the same procedure.

Polycondensation with Diamines. **2** (1.2 g), hexamethylenediamine (4 g), and polyphosphoric acid (10 g) were mixed at 80°C with constant stirring to give a clear solution. The temperature was then raised to 170°C and maintained for 8 h. The polymer was isolated in the usual manner. It does not melt up to 360°C. Under the same condition, *p*-phenylenediamine, benzidine, and 4,4′-oxydianiline were condensed with **2** to give **6b**, **6c**, and **6d**, respectively.

RESULTS AND DISCUSSION

The products reported here are insoluble in all common organic solvents; some of them, **4a-c**, are soluble in the aprotic solvents like DMF, DMSO, DMAc, and NMP while all the polymers **4a-c** and **6a-d** are soluble in concentrated sulfuric acid, *m*-cresol, and formic acid. The nitrogen content of the polymer samples is lower than calculated on the basis of the assumed repeat unit. This has also been observed by other workers [5, 6] and is probably indicative of incomplete decomposition because of the outstanding thermal stability. Due to their poor solubility in common solvents, it was only possible to study solution viscosity in concentrated sulfuric acid. The intrinsic viscosities (Table 1) reveal that the products have fairly high molecular weights.

The IR spectra of these polymers show the characteristic features that correspond to those of the corresponding model compound, 2-methyl-4-

TABLE 1. Characterization of Poly(2-vinyl-3-substituted-4-quinazolone)s

Polymer sample	% N[a]		Density, g/mL	Yield, %	IR (cm^{-1})[b]		Intrinsic viscosity[c] [η], dL/g	Decomposition, temperature,[d] °C
	Found	Calculated			$\nu_{C=O}$	$\nu_{C=N}$		
4a	14.81	16.28	1.35	76	1680	1600	0.32	375
4b	13.78	15.05	1.12	73	1680	1610	0.25	373
4c	9.32	11.29	1.17	78	1675	1605	0.19	370
6a	11.92	12.15	1.21	63	1675	1600	0.38	395
6b	11.86	13.40	1.19	66	1670	1595	0.52	390
6c	9.18	11.34	1.23	58	1665	1590	0.46	380
6d	7.58	10.98	1.14	60	1670	1600	0.40	390

[a]Nitrogen contents based on an average of three results.
[b]KBr pellets.
[c]In concentrated sulfuric acid at 30°C.
[d]From thermogravimetric analyzer, 10°C/min in air.

quinazolone [7]. All the products gave a band around 1680 cm^{-1} characteristic of C=O stretching, a band around 1600 cm^{-1} due to C=N stretching vibration, and bands around 1520 cm^{-1} due to C=C stretching and in-plane bending vibrations of the aromatic moiety. The position of the C=O stretching frequency varied around 1680 cm^{-1} due to structural variation (Table 1).

TC thermograms revealed that the products have thermal stability comparable with that of the reported polyquinazolones [2, 5, 6], starting to degrade around 370°C (Table 1).

All these data support the assumption that the polymeric products have the polyquinazolone structures shown in the formulas.

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