Hemolytic Activity of a Dental Adhesive Monomer (N-methacryloyloxy-5-aminosalicylic acid, MASA) and its Interaction with Phospholipid Liposomes as Studied by NMR and DSC

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N-methacryloyloxy-5-aminosalicylic acid (MASA) has recently been used as an adhesive primer in restorative resin systems. To monitor the biological activity of MASA, we studied changes in NMR-chemical shifts (δ_H) and the differential scanning calorimetry (DSC) phase transition temperature (T_m) of dipalmitoylphosphatidylcholine (DPPC)/MASA liposomes with or without the presence of albumin and collagen. The δ_H and the T_m did not alter significantly and the interaction of MASA with DPPC was found to be small. Hemolytic activity of MASA was markedly smaller than that of the phosphate monomer (MDP) in bonding agents widely used. These findings suggest that using a MASA primer in resin systems has an acceptable biocompatibility for dentin-pulp, involving its adsorption and adhesion to hard tooth tissues.

Key words : MASA, Hemolysis, Phospholipid liposomes

INTRODUCTION

Monomers with carboxylic acid are particularly effective in promoting adsorption and adhesion to cationic surfaces such as the hydroxyapatite in hard tooth tissues¹⁻⁴. Chelating agents containing the polymerizable methacrylate group have been synthesized, and the ability of N-methacryloyloxy-5-aminosalicylic acid (MASA) to promote adhesion between resin system and hard tooth tissues has been studied. The methacrylate group is considered to have a proven sedative effect on pulp, since they are derived from salicylic acid⁵. Recently, MASA has been used as an adhesive primer due to its strong bond between resin and dentin. In clinical applications, a desensitizing effect on hypersensitive dentin has been suggested⁶. Thus, an adhesive MASA primer has been applied to the resin restorative system with an acceptable pulpal response and clinical performance^{7,8}.

To clarify the mechanism of interaction of dental adhesive monomers on biological membranes at a molecular level, we have intensively investigated their interaction with human erythrocytes and with phospholipid liposomes that used as a model for biomembranes^{9,10}. NMR and differential scanning calorimeter (DSC) findings are used to interpret the degree and nature of biological activities caused by the action of monomers^{9,10}.

In the present study, we examined the degree of hemolytic activity caused by MASA as compared with methacryloyloxydecyl dihydrogenphosphate (MDP). Changes in the δ_H and the phase transition temperature (T_m) of L- α -dipalmitoylphosphatidylcholine (DPPC)/MASA liposomes with or without the presence of albumin (or collagen) in D₂O and/or a D₂

O buffer solution were also studied.

MATERIALS AND METHODS

L- α -dipalmitoylphosphatidylcholine (DPPC)* cholesterol (CS)* 3-(trimethylsilyl) propionic acid sodium salts-d₄ (TMSPA)**, dimethylsulfoxide-d₆ (DMSO)**, deuterium oxide(D₂O)**, KD₂PO₄+, and 40% NaOD** were used without further purification. N-methacryloyloxy-5-aminosalicylic acid (MASA) was a gift and was used without further purification. Chemical structure of MASA, DPPC and their numbering systems are shown in Fig. 1. ¹H-NMR of MASA in DMSO-d₆ (The δ_H values are in ppm up- and downfield from DMSO-d₆ (δ_H =2.50ppm) δ_H : 1.94 (3H, s, 2-CH₃), 5.47 (1H broad s, 1Ha), 5.80(1H,broad s, 1Hb), 6.90 (1H, d, J=9.2Hz, 5'H), 7.77 (1H, double d, J=9.2 and 2.4Hz, 6'H), 8.16 (1H, d, J=2.4Hz, 2'H), and 9.72 (1H, s, NH))

Albumin (Human, Fraction V, powder)⁺⁺ and collagen (Type VII, acid soluble from rat tail)* were used without further purification.

The preparation of liposomes, NMR, DSC and hemolysis studies, were identical to those described in our previous papers^{9,10}. The preparation of a D₂O buffer solution is as follows.

Fig. 1 Structure of MASA (N-methacryloyloxy-5-aminosalicylic acid) and DPPC (dipalmitoylphosphatidylcholine), Numbering and small alphabets described for NMR-assignment.

* Sigma Chemical Co., St. Louis, MO, USA. **Merck Chemical Co., Darmstadt, Germany.

+ Kanto Chemical Co., Tokyo, Japan.

++ Tokyo Kasei Chemical Co., Tokyo, Japan.

A solution of 0.1M KD_2PO_4 - D_2O was adjusted to pH 7.03 by addition of 40% NaOD - D_2O solution.

The methyl methacrylate (MMA) and methacryloyloxydecyl dihydrogenphosphate (MDP) used were the same as previously described in this journal⁹.

RESULTS AND DISCUSSION

NMR studies of DPPC/MASA liposomes in a buffer solution

The ^1H -NMR spectra of DPPC liposomes and DPPC/MASA (1:1 molar ratio) liposomes at 52°C, and their chemical shifts (δ_{H}) and chemical shift differences ($\Delta\delta_{\text{H}}$) at 30°C and 52°C in D_2O buffer solution at pH 7.03 are shown in Fig. 2 and Table 1, respectively. Since the spectrum of MASA in DPPC bilayers at 30°C was similar to that at 52°C, only that at 52°C is shown in Fig. 2. The phase transition temperature (T_m) of DPPC/MASA (1:1 molar ratio) liposomes and DPPC liposomes were 35°C and 40.5°C, respectively (see Table 4). From this, it was found that DPPC/MASA liposomes are in a liquid phase at 52°C, above the T_m . Signals of the choline N-methyl (x), the methylene in acyl chains (y), and the terminal methyl (z) group due to DPPC were observed in both DPPC and DPPC/MASA liposomes. Except for those groups, a signal from the N-methylene group (w) was newly observed in DPPC /

Fig. 2 ^1H -NMR spectra of DPPC liposomes and DPPC/MASA liposomes in D_2O buffer solution at pH 7.03 at 52°C.

A: DPPC liposomes, B: DPPC/MASA (1:1 molar ratio) liposomes and numbering: see Fig. 1.

Table 1 ^1H -NMR chemical shifts (δ_{H}) and chemical shift difference ($\Delta\delta_{\text{H}}$) of N-methacryloyloxy-5-aminosalicylic acid (MASA) and dipalmitoylphosphatidylcholine (DPPC) in DPPC/MASA (1 : 1, molar ratio) liposomes in D_2O buffer solution at pH 7.03.

Proton Nos.	MASA		DPPC/MASA		$\Delta\delta_{\text{H}}$	$\Delta\delta_{\text{H}}$	$\Delta\delta_{\text{H}}$
	30°C	52°C	30°C	52°C			
	A	B	C	D			
2CH ₃	1.99 m	2.00 m	1.99	2.00	0.00	0.00	
1Ha	5.54 brs	5.54 brs	5.54	5.54	0.00	0.00	
1Hb	5.80 brs	5.80 brs	5.80	5.81	0.00	0.01	
5'H	6.93 d*	6.94 d#	6.93	6.93	0.00	0.00	
6'H	7.44 dd**	7.45 dd#	7.44	7.46	0.00	0.01	
2'H	7.74 d***	7.76 d# #	7.74	7.76	0.00	0.00	
w NCH ₂			NS	3.67			—
x N ⁺ (CH ₃) ₃				3.24			−0.03
y (CH ₂) ₁₄			NS	1.30			0.00
z CH ₃			NS	0.91			0.00

The δ_{H} value is ppm downfield from TMSPA as used an external reference in D_2O buffer solution. Proton Nos and alphabets (w–z) : see Fig. 1, m : multiplet, brs : broad singlet, d : doublet, dd : double doublet, * : J=8.6Hz, ** : J=2.6Hz and 8.6Hz, *** : J=2.6Hz, # : J=8.9Hz, # # : J=2.6Hz and J=8.9Hz, # # # : J=2.6Hz, NS : no signal, E : DPPC liposomes.

MASA liposomes (see Fig. 2), indicating that the phosphodiester group (O–C–C–N bond) in DPPC was affected by MASA. As an observation consistent with the suggestion, we know a large interaction of MDP with the phosphodiester group⁹, since the phosphodiester group in DPPC serves as an H-bond acceptor¹¹. As shown schematically in Fig. 1, the hydrogen bond between carbonate ions and OH groups in MASA is formed in water and there by H⁺ allowing MASA to easily affect the phosphodiester group (O–C–C–N bond) in DPPC bilayers. However, as shown in Table 1, the δ_{H} values (2-CH₃, 1 Ha, 1Hb, 5'H, 2'H, and 6'H) at 30°C were 0.00 ppm and those at 52°C were 0.00–0.01 ppm, indicating that MASA in the DPPC bilayers is non-shielding and therefore, located in a region close to the surface at both 30°C and 52°C. The interaction of MASA with the DPPC bilayer at pH 7.03 were considered weak.

NMR studies of DPPC/CS/MASA liposomes in D₂O The ^1H -NMR spectra of DPPC/CS/MASA (4 : 1 : 2 molar ratio) liposomes (A) and MASA (B) and δ_{H} and $\Delta\delta_{\text{H}}$ value of their signals in D_2O are shown in Fig. 3 and Table 2, respectively. As shown in Fig. 3 at 30°C, signals in A due to the 2-CH₃, 1Ha, 1Hb, 5'H, 6'H and 2'H of MASA were markedly broader than those of MASA in B. Signals due to the x, y, and z groups of DPPC disappeared. As shown in Table 2, downfield shifts occurred for 1Hb, 2'H, 5'H and 6'H both at 30°C and 52°C. The $\Delta\delta_{\text{H}}$ values of 2'H and 6'H were 0.06–0.07 and 0.06–0.13 ppm, respectively and those of 2CH₃, 1Ha, 1Hb, and 5'H was 0.00–0.02 ppm. Large significant downfield shifts occurred for 2'H and 6'H. The $\Delta\delta_{\text{H}}$ values of MASA in DPPC/MASA (1 : 1 molar ratio) without CS in D_2O were also determined, indicating that large downfield shifts (0.12–0.23 ppm) occurred for 2'H and 6'H. The $\Delta\delta_{\text{H}}$ values of MASA in DPPC/MASA (1 : 1 molar ratio) without CS in D_2O were also determined, indicating that large downfield shifts (0.12–0.23 ppm) occurred for 2'H and 6'H (data not shown). This indicates that the 2'H and 6'H moiety is probably located on the surface of the liposomes without being incorporated into the acyl chains of

Fig. 3 ^1H -NMR spectra and DPPC/CS/MASA liposomes in D_2O at 30°C .

A: DPPC/CS/MASA (4:1:2 molar ratio) liposomes, B: MASA and numbering: see Fig. 1.

Table 2 ^1H -NMR chemical shifts (δ_{H}) and chemical shift difference ($\Delta\delta_{\text{H}}$) of N-methacryloyloxy-5-aminosalicylic acid (MASA) in DPPC/CS/MASA (4:1:2 molar ratio) liposomes in D_2O .

Proton Nos.	MASA		DPPC/CS/MASA		$\Delta\delta_{\text{H}}$ C-A	$\Delta\delta_{\text{H}}$ D-B
	30°C	52°C	30°C	52°C		
	A	B	C	D		
2CH ₃	2.02 m	2.03 m	2.04	2.03	0.02	0.00
1Ha	5.57 brs	5.57 brs	5.58	5.57	0.01	0.00
1Hb	5.84 brs	5.83 brs	5.85	5.85	0.01	0.02
5'H	6.98 d*	6.98 d#	7.00	6.99	0.01	0.01
6'H	7.49 dd**	7.50 dd# #	7.55	7.63	0.06	0.13
2'H	7.79 d***	7.80 d# # #	7.85	7.87	0.06	0.07

The δ_{H} value is ppm downfield from TMS-PA as used an external reference in D_2O . Proton Nos: see Fig. 1, m: multiplet, brs: broad singlet, d: doublet, dd: double doublet, *: $J=8.6\text{Hz}$, **: $J=2.6\text{Hz}$ and 8.6Hz , ***: $J=2.6\text{Hz}$, #: $J=8.9\text{Hz}$, # #: $J=2.6\text{Hz}$ and $J=8.6\text{Hz}$, # # #: $J=2.6\text{Hz}$.

DPPC. This finding was different from that described above in a D_2O buffer (Table 1). This appears to be due to the fact that most of MASA changed to its un-ionized state in D_2O and, thus, the CONH group of MASA which is a hydrophilic moiety expands in water. Thereby, the 2'H and 6'H group was located on the surface of the liposomes.

Table 3 Chemical shift differences ($\Delta\delta_{\text{H}}$) between DPPC/MASA (1:1 molar ratio) liposomes in the presence of albumin (or collagen) and DPPC/MASA (1:1) liposomes in D_2O buffer solution at pH 7.03.

Proton Nos.	DPPC/MASA liposomes/albumin		DPPC/MASA liposomes/collagen	
	30°C	52°C	30°C	52°C
2CH ₃	0.01	0.00	0.02	0.01
H1a	0.00	0.00	0.02	0.01
H1b	0.01	0.00	0.02	0.01
5'H	0.01	0.01	0.01	0.02
6'H	0.02	0.00	0.02	0.01
2'H	0.01	0.01	0.01	0.01
x (CH ₃) ₃		0.00		0.00
z terminal CH ₃		0.00		0.01
y (CH ₂) ₁₄		0.00		0.01

$\Delta\delta_{\text{H}} = \delta_{\text{H}}$ of DPPC/MASA liposomes/albumin (or collagen) $-\delta_{\text{H}}$ of DPPC/MASA liposomes, Proton Nos and alphabets (x-z): see Fig. 1, and each of an 1.5% albumin and of 0.1% collagen was added to DPPC/MASA liposomes.

NMR studies of DPPC / MASA liposomes in the presence of albumin and collagen

Monomers containing such substituents as hydroxy and carboxy groups may interact with albumin and collagen. As shown in Table 3, albumin or collagen was added to the DPPC/MASA (1:1 molar ratio) liposomes. Chemical-shift difference values ($\Delta\delta_{\text{H}} = \delta_{\text{H}}$ of DPPC/MASA liposomes/albumin (or collagen) $-\delta_{\text{H}}$ of DPPC/MASA liposomes) at 30°C and 52°C were 0.00–0.02 ppm, respectively, indicating that the addition of protein to the liposomes did not significantly alter the chemical shifts of MASA. Within allowances for experimental error (δ_{H} : 0.02ppm), the $\Delta\delta_{\text{H}}$ values of the choline N-methyl, the methylene of (CH₂)₁₄ and the terminal methyl group were practically unaltered as compared with DPPC/MASA liposomes (minus 0.01–0.01). The interaction of MASA in lipid bilayers with albumin (or collagen) in D_2O buffer solution at pH 7.03 was considered small.

DSC studies The phase transition temperature (T_{m}) and the phase transition profile (H/HHW) of various liposome systems involving DPPC are shown in Table 4. The T_{m} of DPPC liposomes was 40.5°C, while that of DPPC / MASA liposomes in the molar ratio of 1:1 and 2:1 was 36–36.5°C with a broadening of the DSC peak (H/HHW=18–20). The T_{m} of DPPC induced by MASA shifted lower 4–4.5°C. The T_{m} of the various liposome systems in the presence of albumin and collagen are shown in Table 4. The T_{m} of DPPC / MASA (1:1)+ albumin or collagen shifted lower by 1.5–2°C as compared with original DPPC/MASA liposomes, but that in the molar ratio of 2:1 was unaltered. Since allowance for experimental error in this study was 0.5°C, the change in the T_{m} described above was not large. The T_{m} of DPPC liposomes in the presence of proteins was unaltered as compared with that of the original DPPC liposomes. These findings suggest that the interaction of DPPC-MASA was larger than that of MASA-albumin (or collagen) and the interaction between DPPC and protein was small. The H/HHW value of DPPC/MASA liposomes reduced in the presence of albumin (or collagen). This indicates that the addition of protein had a relatively large effect on cooperativity of the phase transition of DPPC/MASA liposomes i. e., the surface

Table 4 Changes in phase transition temperature (T_m) and transition profile value (H/HHW) of various DPPC-liposome system

Liposome system (molar ratio) [#]	T _m °C	H/HHW ^{##}
DPPC/MASA (1 : 1)	36.5	20
DPPC/MASA (1 : 1)	36	20
DPPC/MASA (1 : 1) + Albumin	35	7
DPPC/MASA (1 : 1) + Albumin	35.5	7
DPPC/MASA (1 : 1) + Collagen	34.5	7
DPPC/MASA (1 : 1) + Collagen	35	6
DPPC/MASA (2 : 1)	36	18
DPPC/MASA (2 : 1)	36.5	18
DPPC/MASA (2 : 1) + Albumin	35.5	7
DPPC/MASA (2 : 1) + Albumin	36	5
DPPC/MASA (2 : 1) + Collagen	36	5
DPPC/MASA (2 : 1) + Collagen	36	3
DPPC/MASA (1 : 1)*	35	24
DPPC/MASA (1 : 1)*	35	24
DPPC*	40.5	100
DPPC + Albumin*	40	94
DPPC + Collagen*	40.5	83

DPPC: dipalmitoylphosphatidylcholine, MASA : N-methacryloyloxy-5-aminosalicylic acid, [#] : in D₂O, ^{##} : Height/Half-Height Width of DSC main peak (as DPPC liposomes are to 100), * : in a D₂O buffer solution at pH 7.03, 70 mM DPPC in a solution, 1.5% albumin, and 0.1% collagen.

Table 5 Percentage of hemolysis caused by N-methacryloyloxy-5-aminosalicylic acid (MASA), methyl methacrylate (MMA) and methacryloyloxydecyl dihydrogen phosphate (MDP) at 37°C

Additives*	Concentration mM	Reaction time min	Hemolysis % Mean (SD)
MASA	19	50	2.1 (0.5)
MMA	19	50	1.0 (0.1)
MDP ⁺	19	10	DN

An 1% human erythrocyte in phosphate buffer solution at pH 7.0. DN : Denaturalization of hemoglobin after hemolysis, * : diluted in ethanol and + : diluted in diethylether, and mean (SD) : five samples.

adsorption of protein molecules did partly occur. However, it is clear that the interaction was quite small.

Hemolysis studies Hemolytic activity of MASA, MMA and MDP are shown in Table 5. MASA's degree of hemolysis at 19mM was 2.1%. That of MDP at 19mM was not calculated after 10min because denaturalization of hemoglobin occurred after strong hemolysis. From this, it was concluded that the hemolytic activity of MASA was markedly weaker than that of MDP, indicating that MDP has stronger surface-active action and has a higher acidity than

MASA. MMA's degree of hemolysis at 19mM was 1.0%. Hemolytic activity of MMA increases when it reaches a high concentration (above 100mM) due to its low hydrophobicity¹²⁾. These findings suggest that the biological effect of MASA on pulp is much weaker than that of MDP. The recently reported findings that the biocompatibility of the MASA resin-system is better than that of the MDP-resin system^{7,8)} are also supported by these findings.

CONCLUSIONS

The δ_H and $\Delta\delta_H$ of MASA in DPPC/MASA liposomes, with or without the addition of albumin and collagen, in D₂O buffer solution at pH 7.03 were determined by NMR spectroscopy. The $\Delta\delta_H$ of MASA was unaltered under these conditions. Addition of albumin or collagen to DPPC/MASA liposomes did not have a large effect on the T_m, but did have a relatively large effect on the H/HHW value. Hemolytic activity of MASA was markedly smaller than that of MDP. Therefore, using a MASA primer for resin restorations has an acceptable biocompatibility for the dentin-pulp system, involving its adsorption and adhesion to hard tooth tissues.

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歯科用接着性モノマー (N-methacryloyloxy-5-aminosalicylic acid, MASA) の 溶血性及びりん脂質リポソームとの相互作用の NMR, DSC 研究

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最近 MASA は接着性プライマーとしてレジン修復システムに用いられている。MASA の生物学的活性をモニターするため、アルブミン及びコラーゲン存在下ジパルミトイルホスファチジルコリン (DPPC) / MASA リポソーム系の NMR ケミカルシフト (δ_H) 及び DSC 相転移温度 (T_m) を研究した。その結果、蛋白の存在はリポソームの δ_H 及び T_m に変化を与えなかった。また

MASA とりん脂質 (DPPC) との相互作用も小さいことが明らかになった。MASA の溶血性はボンディング剤としては広く用いられているりん酸モノマー (MDP) に比べ著しく小さかった。以上の所見は MASA プライマーは象牙質-歯髄システムに対して生体適合性があることを示唆した。

コンポジットレジン修復時に生ずる窩縁部エナメル質微小亀裂に及ぼす 臨床的因子に関する研究

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本研究では、コンポジットレジン修復時に生ずるエナメル質微小亀裂を、歯牙表層及び縦断面において、実体顕微鏡及び SEM を用いて観察した。さらに、エナメル質微小亀裂の発生に及ぼす臨床的因子、すなわち、重合方式、窩縁形態及び研磨時期の影響について検討した。

エナメル質微小亀裂は、ベベルを付与せず充填直後に研磨を行った 1 級及び 5 級の光重合型コンポジットレジン修復物において、全例に認められた。エナメル質微小

亀裂は、窩洞辺縁にほぼ平行で、窩洞辺縁より 0.01~0.3 mm 離れた部位に観察された。エナメル質微小亀裂の発生は、ノン・ベベル窩洞で充填後 10 分あるいは 24 時間後に研磨を行った群において、光重合型のほうが化学重合よりも著しかった。エナメル質微小亀裂の発生は、ベベルの付与及び研磨時期を遅らせることにより減少した。

歯の形状の三次元計測 (第 11 報)

— 咬合を考慮したクラウン形状設計のための CAD について —

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[6] 臼歯のクラウン補綴のための形状設計の CAD 化につき研究した。[6] にクラウン用の支台歯形成がされている, [5]~[7] 臼歯の石こう模型と、修復用に用いる [6] 完全歯の計測を行った。

クラウンデータは [6] の支台歯に適合し [5]~[7] の隣接歯と

の調整を行った。その後, [6] のデータの座標は [6] 支台歯のそれに変換した。適合したクラウンの端部は, [6] 支台歯の辺縁と結合した。

さらに, 対合歯との咬合関係を FGP 法を応用して調整した。FGP は, バイトワックスに記録し計測した。